INTERLABORATORY STUDY 89-5

EXTRACTABLE ORGANIC PARAMETERS IN REAGENT WATER

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1 SUMMARY OF INTERLABORATORY STUDY 89-5

Interlaboratory Study 89-5 was the third of an on-going program of laboratory performance management studies initiated by the Quality Assurance Office, Laboratory Services Branch of Environment Ontario. It assesses the performance of participating laboratories for the analysis of selected organic parameters (Table II, Section 2.1) in spiked reagent water. Fifteen laboratories (government and commercial) agreed to participate in this study. Results were received from twelve participants.

The parameters were chosen from three different MISA analytical Test Groups (1). The results for each parameter group were interpreted separately.

The results demonstrate variable performance among laboratories. Some participants were able to achieve good recovery (80-110%) of the target parameters across the scan for the Base/Neutral Extractables (MISA Group 19) and the Neutral Chlorinated Extractables (MISA Group 23). Other laboratories demonstrated biases due to differences in calibration standards or analytical procedures. Some of the laboratories experienced some laboratory contamination with phthalates. These compounds are commonly used as plasticizers and are common laboratory contaminants.

The results for the Acid Extractables (MISA Group 20) were more variable, with mean recoveries ranging from 31-113%. Very few of the participants were able to achieve consistent performance for this parameter group.

Differences in standards may result in consistent over- or under-recovery across the scan. Differences in analytical procedures may produce a variety of effects, including over- or under-recovery of specific parameters, patterns of increasing or decreasing recovery across the scan, and variability of recovery of low level spikes versus high level spikes. It is desirable that laboratories demonstrating some of these problems strive to correct these difficulties. Individual laboratory performance is reviewed for the above performance criteria and some suggestions for areas of improvement are provided. Future interlaboratory studies that include the analysis of the Base/Neutral, Acid, and Neutral Chlorinated Extractable Organics will attempt to monitor improvements in performance by the participants.

2 SUMMARY OF RESULTS

Due to the large number of parameters included in this interlaboratory study, this report does not attempt to assess the between-laboratory performance of each individual parameter. A review of the range of results given by the Minimum and Maximum values in Tables 1,3, and 5, Appendix 1, indicates that the results may vary by an order of magnitude. However, as there were only two spiked samples to each participant, there is insufficient data to assess the analytical characteristics of each individual parameter. Future studies will attempt to assess the analytical characteristics of individual parameters by submitting more samples to the participants and including duplicate analyses. An overview of the interlaboratory performance for each MISA analytical Test Group is given in Section 5.

The following table summarizes each individual laboratory's performance. The description for the ranking procedure for the interanalyte variability and interanalyte recovery is given in Section 4.4. A detailed review of each participant's performance, with some recommendations for areas of improvement, is given in Section 5.

TABLE I - SUMMARY OF PARTICIPANTS' PERFORMANCE

NOTE: See Section 4.4 for detailed description of ranking procedure.

LAB CODE	MISA TEST GROUP	PERFORMANCE
2001	Base/Neutrals (19)	- Excellent within-laboratory interanalyte repeatability - Satisfactory performance for interanalyte variability - Low bias relative to design value - Results within one standard deviation of interlaboratory mean and median
	Acids (20)	- Satisfactory within-laboratory interanalyte repeatability - Very variable interanalyte performance - Good recovery relative to the design value - Results were higher than interlaboratory mean and median, sometimes by more than one standard deviation; results within one standard deviation of design values
	Neutral Chlorinated (23)	- Results outside upper within-laboratory precision limits due to low recovery of one sample - Very variable interanalyte performance - Very low bias relative to design value - Variable performance relative to the interlaboratory mean and median; several parameters lower by more than one standard deviation
2002	Base/Neutrals (19)	- Some inconsistencies regarding within- laboratory interanalyte repeatability - Satisfactory performance for interanalyte variability - Good recovery relative to design value; some parameters indicate slight low bias - Most results within one standard deviation of interlaboratory mean and median

LAB CODE	MISA TEST GROUP	PERFORMANCE
2002	Acids (20)	- Satisfactory within-laboratory interanalyte repeatability - Satisfactory performance for interanalyte variability with one outlier - Recovery of parameters in two groups, one group has slight low bias and other group very low bias - Results within one standard deviation of interlaboratory mean and median except for 2,4-Dichlorophenol
	Neutral Chlorinated (23)	- Satisfactory within-laboratory interanalyte repeatability - Satisfactory performance for interanalyte variability - Excellent interanalyte recovery - Results were higher than interlaboratory mean and median, sometimes by more than one standard deviation; results within one standard deviation of design values
2003	Base/Neutrals (19)	- Some inconsistencies regarding within-laboratory interanalyte repeatability; some problem parameters - Variable interanalyte performance - Good recovery relative to the design values with some parameters slight low bias - Consistent recovery across the scan for the low spike; pattern of increasing recovery across the scan for the high spike - Most results within one standard deviation of interlaboratory mean and median
	Acids (20)	- Consistent within-laboratory interanalyte repeatability - Very variable interanalyte performance - Biased low relative to design value - Results lower than the interlaboratory mean and median, but most were within one standard deviation
	Neutral Chlorinated (23)	- Satisfactory within-laboratory interanalyte repeatability - Variable interanalyte performance - Biased low relative to design value - Results lower than the interlaboratory mean and median, but most were within one standard deviation
2004	Base/Neutrals (19)	- Poor within-laboratory interanalyte repeatability - Satisfactory performance interanalyte variability - Low bias for the low spike and a slight high bias for the high spike - Results for low spike within one standard deviation of the interlaboratory mean and median; results for high spike were high relative to interlaboratory mean and median
	Acids (20)	- Poor within-laboratory interanalyte repeatability - Interanalyte performance very variable - Very variable recovery relative to the design value - Variable performance compared to the interlaboratory mean and median; many parameters differ by more than one standard deviation

LAB CODE	MISA TEST GROUP	PERFORMANCE
2004	Neutral Chlorinated (23)	- Good within-laboratory interanalyte repeatability - Very variable interanalyte performance - One group of parameters with a low bias and the second group with a very low bias relative to design value - Results low compared to the interlaboratory mean and median
2005	Base/Neutrals (19)	- Some parameters demonstrate satisfactory within-laboratory interanalyte repeatability, but may results are outside precision limits - Very variable interanalyte performance - Good recovery for some parameters but low bias for many parameters - Results variable compared to the interlaboratory mean and median; several parameters differ by more than one standard deviation
	Acids (20)	- Poor within-laboratory interanalyte repeatability - Very variable interanalyte performance - Variable recovery relative to the design value; tendency to low bias - Results variable compared to the interlaboratory mean and median
	Neutral Chlorinated (23)	- Good within-laboratory interanalyte repeatability - Satisfactory performance for interanalyte preformance - Good recovery relative to the design value; slight low bias for some parameters - Consistent results relative to the interlaboratory mean and median
2006	Base/Neutrals (19)	- Satisfactory within-laboratory interanalyte repeatability - Satisfactory peformance for interanalyte variability - Very low bias relative to the design value - Results biased low relative to the interlaboratory mean and median
	Acids (20)	- Satisfactory within-laboratory interanalyte repeatability - Satisfactory performance for interanalyte variability for most parameters; some parameters not detectted in low spike - Low bias relative to the design value - Results biased low relative to the interlaboratory mean and median
	Neutral Chlorinated (23)	- Good within-laboratory interanalyte repeatability - Variable interanalyte performance - Biased low relative to design value - Results biased low relative to the interlaboratory mean and median

LAB CODE	MISA TEST GROUP	PERFORMANCE
2007	Base/Neutrals (19)	- Within-laboratory interanalyte repeatability satisfactory, but is biased low outside the precision limits due to low recovery of high spike - Satisfactory performance for interanalyte variability - Low bias for low spike, very low bias for high spike, relative to design value - Results biased low relative to the interlaboratory mean and mean
	Acids (20)	- Good within-laboratory interanalyte repeatability - Variable interanalyte performance - This laboratory noted that they used an old calibration standard for this scan; results were biased low relative to design values and may be attributed to old standard - Results were biased low relative to the interlaboratory mean and median; may be attributed to old standard as noted above
	Neutral Chlorinated (23)	- Within-laboratory interanalyte repeatability good, but is biased low outside the precision limits due to low recovery of high spike - Excellent performance for interanalyte variability - Results biased low relative to the design values - Good agreement with the interlaboratory mean and median for the low spike; high spike was low relative to the interlaboratory mean and median
2008	Base/Neutrals (19) Acids (20) Neutral Chlorinated (23)	- Technician error occurred during extraction of their samples therefore chromatography was severely affected; not possible to assess performance
2010	Base/Neutrals (19)	- Variable within-laboratory interanalyte repeatability - Variable interanalyte performance - Good recovery relative to the design value - Results biased high relative to the interlaboratory mean and median; many parameters differ by more than one standard deviation but are within one standard deviation of the design values
	Acids (20)	- Poor within-laboratory interanalyte repeatability - Very variable interanalyte performance - Variable recovery relative to design value - Results were slightly high compared to the interlaboratory mean and median; some parameters in the high spike differed by more than one standard deviation
	Neutral Chlorinated (23)	- Good within-laboratory interanalyte repeatability - Excellent performance for interanlyte variability - Slight low bias relative to design value - Good agreement with the interlaboratory mean and median

LAB CODE	MISA TEST GROUP	PERFORMANCE
2011	Base/Neutrals (19)	- Satisfactory within-laboratory interanalyte repeatability - Satisfactory performance for interanalyte variability - Good recovery relative to the design value - Good agreement with the interlaboratory mean and median for most parameters; some values on the high side
	Acids (20)	- Satisfactory within-laboratory interanalyte repeatability - Very variable interanalyte performance - Variable recovery relative to the design value - Slight pattern of decreasing recovery across the scan - Good agreement with the interlaboratory mean and median for most parameters
	Neutral Chlorinated (23)	- Results withdrawn after initial tables of results distributed to participants; interlaboratory study samples were mixed-up with other samples by the laboratory
2012	Base/Neutrals (19)	- Good within-laboratory interanalyte repeatability for some parameters, variable precision for others - Variable interanalyte performance - Samples contaminated with Bis(2-ethylhexyl)phthalate, including the lab blank - Low bias relative to design value - Pattern of decreasing recovery across the scan for the low spike but consistent recovery across the scan for the high spike - Good agreement with interlaboratory mean and median except for problem parameter noted above
	Acids (20)	- Good within-laboratory interanalyte repeatability - Satisfactory performance for interanalyte variability - Good recovery relative to design values with slight low bias for some parameters - Good agreement with the interlaboratory mean and median
	Neutral Chlorinated (23)	- Variable within-laboratory interanalyte repeatability - Satisfactory interanalyte performance for low spike; variable interanalyte performance for high spike - Low bias relative to the design value - Consistent recovery across the scan for the low spike; pattern of increasing recovery across the scan for the high spike - Good agreement with the interlaboratory mean and median
2013	Base/Neutrals (19)	- Satisfactory within-laboratory interanalyte repeatability - Satisfactory performance for interanalyte variability - Slight low bias relative to design value for low spike; good recovery for high spike - Good agreement with the interlaboratory mean and median for most parameters

LAB CODE	MISA TEST GROUP	PERFORMANCE
2013	Acids (20)	- Poor within-laboratory interanalyte repeatability - Very variable interanalyte performance - Variable recovery relative to the design values; 4,6-Dinitro-o-Cresol biased considerably high and excluded from statistical calculations - Results demonstrate a pattern of increasing recovery across the scan - Variable performance relative to the interlaboratory mean and median
	Neutral Chlorinated (23)	- Variable within-laboratory interanalyte repeatability - Very variable interanalyte performance - Variable recovery relative to design value - Parameters appeared as two different groups across the scan, with a sudden shift in recovery taking place - Some parameters had good agreement with the interlaboratory mean and median while others differed by more than one standard deviation

3 INTRODUCTION

Interlaboratory performance studies are conducted to assess the comparability of data among different laboratories. These studies help in the identification of biases, precision and accuracy problems. Participation in such studies helps in improving individual laboratory performance and maintaining performance standards. The Quality Assurance Office, Laboratory Services Branch (LSB), Environment Ontario has instituted an on-going program of interlaboratory studies to assess and enhance the performance of environmental laboratories providing analytical services.

This study was designed to assess the analytical variability between laboratories for the analysis of base/neutral, acid, and neutral chlorinated extractable organics. Parameters were chosen from analytical test groups listed in the MISA (Municipal and Industrial Strategy for Abatement) General Regulation (1). Participants were requested to use methods which conformed to the MISA analytical principles and protocols given in the General Regulation (1).

Fifteen laboratories (government and commercial) agreed to participate in this study. Results were received from twelve participants. A list of participants is included in Appendix 2. Each participant was assigned a unique identification code to maintain confidentiality.

A set of three samples was distributed to each of the fifteen participants. The samples consisted of a reagent water blank and two reagent water samples fortified with subsets from MISA Test Groups 19, 20, and 23 (1). Sections 4.1 and 4.2 describe sample preparation and distribution. Section 4.3 describes analytical methodology requirements. Section 4.4 describes data handling. Final results and individual participant's performance are discussed in Section 5.0.

4 PROCEDURE

4.1 Preparation of Samples

A combined Spiking Solution was prepared in deionized distilled water. Stock solutions consisted of four different organic mixtures from the US-EPA and one in-house prepared ampoule. Table II lists the parameters present in each ampoule that are part of MISA Test Groups 19, 20 and 23. The EPA ampoules contained some additional compounds not listed, that are not part of these MISA Test Groups.

Deionized distilled water was used for the sample matrix. Care was taken that no plastic materials came into contact with the water when transferring to one litre amber sample bottles. To each bottle, 800 mL of water was added by weight (800 g ±2 g). Unspiked bottles were labelled "EXTA". The appropriate amount of Spiking Solution was dispensed into the bottles for the low and high spikes. The low spike was labelled "EXTB" and the high spike was labelled "EXTC".

TABLE II - PARAMETER LIST

EPA GC/MS BASE NEUTRAL I WP 286	Bis(2-chloroethyl)ether Nitrosodi-n-propylamine Bis(2-Chloroethoxy)methane 1,2,4-Trichlorobenzene Hexachlorobutadiene 2-Chloronaphthalene 2,6-Dinitrotoluene 2,4-Dinitrotoluene Hexachlorobenzene Phenanthrene Di-n-butyl Phthalate Pyrene Benzo(a)anthracene Benzo(k)fluoranthene
EPA GC/MS BASE NEUTRAL II WP 586	Hexachloroethane Naphthalene Acenaphthene Fluorene Anthracene Fluoranthene Butyl benzyl Phthalate Chrysene Bis(2-Ethylhexyl)phthalate Benzo(b)fluoranthene Benzo(a)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
EPA GC/MS BASE NEUTRAL III WP 186	1,2,3-Trichlorobenzene 1,2,4-Trichlorobenzene 1,2,4,5-Tetrachlorobenzene 1,2,3,4-Tetrachlorobenzene
EPA PHENOLS WP 985	4-Chloro-3-methylphenol 2-Chlorophenol 2,4-Dichlorophenol 2,4-Dimethylphenol 2-Methyl-4,6-dinitrophenol 4-Nitrophenol Pentachlorophenol Phenol 2,4,6-Trichloroohenol
PHNL02 (In-house ampoule)	2,3,4,5-Tetrachlorophenol 2,3,4,6-Tetrachlorophenol 2,3,5,6-Tetrachlorophenol 2,4,5-Trichlorophenol 2,3,4-Trichlorophenol p-Cresol

4.2 Sample Distribution

Prior to sample preparation, a Letter of Notification was sent to each participating laboratory. Written confirmation of participation was required. A list of participating laboratories is given in Appendix 2.

A set of three samples for each participating laboratory was packaged in a cardboard box and shipped via Purolator courier on May 2, 1989.

4.3 Analytical Methodology

Participants were requested to analyze the samples using routine in-house methods that complied with the principles and protocols outlined in Schedule 3 of the MISA General Regulation (1). Participants were not required to provide the details of their analytical procedure.

4.4 Data Handling

Results were submitted to the Quality Assurance Office, LSB in written form. All data were manually entered by laboratory code into an electronic spreadsheet. Blank spaces were left when a laboratory did not report results for a specific parameter that was present in the spiking material (i.e. the participant did not have the appropriate calibration standard). If the result was reported as <MDL or ND, a "0" was entered if the MDL was less than the design value. If the MDL was greater than the design value and <MDL was reported, then the MDL value (if known) was entered.

In several different cases, participants reported a combined value for two (or more) parameters that co-eluted. For the purposes of statistical calculations, the total result was evenly divided between the co-eluting parameters.

The final percent participation was 80%.

Interlaboratory variability was determined by calculating the mean, median, and standard deviation. The minimum and maximum values were identified to give an indication of the range of results. Outliers were not removed from the data set when calculating between-laboratory variability, except for the results from Laboratory 2013 for 4,6-Dinitro-o-cresol. See Section 5.2 for a detailed explanation.

The percent recovery for each parameter was calculated based on the design value of the spiking material. These results are tabulated in Appendix 1.

To assess each laboratory's performance across the scan, bar graphs of the recovery for each parameter from each participating laboratory were prepared and are included in Appendix 1. The different MISA test groups were divided into separate graphs. Parameters are arranged from left to right in approximate order of gas chromatographic elution. This is based on a SPB-5 capillary column for the Base/Neutral Extractables (MISA Test Group 19), a DB-5 capillary

column for the Acid Extractables (MISA Test Group 20), and a DB-1 capillary column for the Neutral Chlorinated Extractables (MISA Test Group 23). All results are presented as percent recovery of the design value. Outliers were not deleted.

To assess within-laboratory performance based on interanalyte repeatability and bias, the paired results, converted to percent recovery, for samples EXTB and EXTC were plotted for each participant using Youden's two-sample technique (2). Separate plots were prepared for each of the MISA Test groups (i.e. separate plots for the Base/Neutrals, Acids, and Chlorinated Neutrals). A 45° line was drawn between the origin and the target value (100%). Precisely biased data is expected to lie on or close to this line. Tolerance limits of ±20% were also drawn on the graphs. This amount of variation for organic analysis should be achievable by all participants. These graphs are included in Appendix 1.

Using the set of graphs described above, individual laboratory performance was assessed for interanalyte variability and interanalyte bias. The different MISA Test Groups were divided into separate graphs. Interanalyte repeatability was ranked according to how the grouping of data points clustered in a circle: radius of ±10% - excellent; radius of ±20% - satisfactory; radius of ±30% - variable; radius of >30% - very variable. The location of the centre of the group of data along the 45° line determined a ranking for interanalyte bias. The following criteria was used to rank performance for recovery: centre at 100% - excellent; centre at 90-110% - satisfactory; centre at 75-85% - slight low bias; centre at 50-70% - low bias; centre <50% - very low bias. There were no laboratories in this study whose data centred around a recovery of >110%.

5 RESULTS AND DISCUSSION

Three laboratories who received samples did not report any results. One laboratory received sample EXTC with the lid off and 100 mL of solution remaining in the bottle. An extra sample was shipped to that participant.

The complete set of reported results (Tables 1, 3 and 5, Appendix 1) was sent to the participants for review in September 1989. Laboratory 2011 requested that their results for the Neutral Chlorinated Extractables (MISA Group 23) be withdrawn from the data set, as the interlaboratory study samples had been mixed with another set of samples. Their results are included separately in Table 5, but are not part of the statistical calculations. Additional information provided by each participant is included with the review of their results.

5.1 Base/Neutral Extractables (MISA Group 19)

OVERVIEW OF INTERLABORATORY PERFORMANCE

The graphical representation of the results arranged in elution order (Figures 1-8) demonstrate that the majority of laboratories perform consistently across the scan. None of the laboratories demonstrate a pattern of increasing recovery across the scan, a phenomenon noted in a previous study (Interlaboratory Study 88-1) involving this parameter group (3).

The previous study (3) also identified the reverse pattern of decreasing recovery across the scan. The results from Laboratories 2001, 2005, 2010, and 2012 for Sample EXTB in this study have lower recoveries for the parameters eluting during the final third of the scan. However this pattern is not displayed for Sample EXTC. With only two samples from each participant, it is not possible to determine if the laboratory's extraction procedure or the temperature program of the gas chromatograph caused the lower recoveries of the parameters eluting at the end of the scan. The laboratories demonstrating this pattern for Sample EXTB should check their in-house QC program to determine if this is a frequent problem or attributable to random error.

The interlaboratory mean for all parameters, except Bis(2-ethylhexyl)phthalate in both samples, and Bis(2-Chloroethoxy)methane in EXTC, was lower than the design value. For Sample EXTB the interlaboratory mean was 1.0-1.5 ppb lower than the design value (16-25% lower). For Sample EXTC the interlaboratory mean was 5.0-6.0 ppb lower than the design value (23-28% lower).

The interlaboratory median for all parameters, except Bis(2-ethylhexyl)phthalate in Sample EXTC, was lower than the design value. For Sample EXTB the interlaboratory median was 1.05-2.5 ppb lower than the design value (18-42% lower). For Sample EXTC the interlaboratory median was 2.95-7.8 ppb lower than the design value (14-37% lower).

Some of the participants exhibited a low bias relative to the design value and to the interlaboratory mean and median. This was particularly noticeable for Laboratory 2006. Laboratory 2007's results were low relative to the design value, but were very close to the interlaboratory mean and median.

None of the participants demonstrated a high bias relative to the design values. Some of the laboratories did have results that were biased high relative to the interlaboratory mean and median. However these laboratories' results were very close to the design value. This suggests that the differences between the laboratories are due to differences in standards, rather than differences in analytical procedures.

Some individual parameters were over-recovered, most commonly the different phthalates. Bis(2-ethylhexyl)phthalate and Di-n-butyl phthalate were the only two parameters detected in the unspiked sample (EXTA). This group of compounds are used as plasticizers and are common laboratory contaminants.

The Youden two-sample plots for each individual laboratory (Figures 9-20) demonstrate that approximately 75% of the laboratories are able to maintain within-laboratory interanalyte variability of ±20%. Individual parameters may be a problem for an individual laboratory.

Due to the large number of parameters included in MISA Group 19, this report does not attempt to assess the between-laboratory performance of individual parameters, other than as noted above regarding phthalates. With only two spiked samples analyzed by each participant, there is insufficient data to assess the analytical characteristics of each parameter. Future studies will attempt to assess the analytical characteristics of individual parameters by submitting more samples to the participants and including duplicate analyses.

The following sections review individual laboratory performance. Performance was assessed according to within-laboratory interanalyte repeatability, percent recovery relative to the design value (interanalyte bias), performance across the scan, and percent recovery relative to the interlaboratory mean and median.

LABORATORY 2001

The samples for this scan were analyzed using GC/MS. The results from this laboratory demonstrated consistent recovery relative to the design value for most parameters. They did not detect 2-Chloronaphthalene in both of the spiked samples. However they did report results for 1-Chloronaphthalene. Participants were not required to provide the name of the GC column that they used, so it is not known whether the two isomers elute close together, and were mis-identified in these samples.

The results demonstrate excellent within-laboratory interanalyte repeatability (Figure 9). The majority of the paired results are close to the target line of precision and all results are within the precision limits.

Interanalyte variability is satisfactory, as the cluster of data points is within a radius of ±20%.

The percent recovery for most parameters is between 60-80%, relative to the design value (Table 2, Appendix 1). The centre point of the data points is approximately 70% (Figure 9), indicating a low bias. These results suggest that the calibration standard for this laboratory is more concentrated than the design value, resulting in the low bias.

The performance across the first two thirds of the scan is consistent for both the low and high spikes (Figures 1 and 5, Appendix 1). A slight decrease in recovery for the parameters eluting during the final third of the scan is noted for both samples. This last group includes several polyaromatic hydrocarbons (PAH's) that are sensitive to a poor injection into the gas chromatograph, thereby reducing their recovery. The low recovery may also be due to interferences from the clean-up step (if used; this information was not required to be submitted by the participants).

Laboratory 2001 demonstrated consistent performance relative to the interlaboratory mean and median. Only one parameter, Chrysene, in Sample EXTB differed from the interlaboratory mean by more than one standard deviation (Table 1, Appendix 1). All the results for Sample EXTC are within one standard deviation of the interlaboratory mean and median.

LABORATORY 2002

This laboratory reported a combined result for Benzo(b)fluoranthene and Benzo(k)fluoranthene. The total result was evenly divided between the two isomers for the purposes of statistical calculations.

The results demonstrate some inconsistencies for within-laboratory interanalyte repeatability (Figure 10). The majority of results are close to the line of target precision. The outlying points indicate some problem parameters that are discussed in detail below.

The majority of data points are within a radius of ±20%, demonstrating satisfactory performance for interanalyte variability.

This laboratory demonstrated good recovery relative to the design value for most parameters. The majority of parameters have recoveries between 80-100% for both spiked samples (Table 2, Appendix 1). Two parameters were over-recovered in Sample EXTB, Bis(2-chloroethoxy)methane and Naphthalene. This may be due to random unidentified laboratory contamination, as the results for Sample EXTC were not affected. One parameter, Benzylbutylphthalate, appears to have been difficult to recover by this laboratory. In Sample EXTB the recovery was 30% and in Sample EXTC the recovery was 29%. Phthalates are common laboratory contaminants. This laboratory may have had a high laboratory blank result for this parameter and by subtracting that value from the day's results, over-corrected the interlaboratory study results. Participants were not required to submit their daily QC values with the interlaboratory study results. The centre of the data points (Figure 10) is approximately 85%. This indicates a slight low bias relative to the design value.

The results across the scan are consistent and do not demonstrate any patterns (Figures 1 and 5). Problem parameters were noted above.

The problem parameters noted above differed from the interlaboratory mean by more than the standard deviation. Most of the other results are within one standard deviation of the interlaboratory mean median. However the results for Bis(2-chloroethyl)ether are higher than the interlaboratory mean and median by more than one standard deviation, for both spiked samples. Greater variability are observed in the low spike (Sample EXTB), as the results for Chrysene, 2,4-Dinitrotoluene, and 2,6-Dinitrotoluene are greater than the interlaboratory mean and median by more than one standard deviation. These results indicate better agreement with the design values for these specific parameters, than that achieved by most of the participants.

LABORATORY 2003

The results demonstrate some inconsistencies for within-laboratory interanalyte repeatability (Figure 11). Most paired results are within the precision limits. Several points are outside the upper precision limits. The parameters that are over-recovered are discussed below.

The data pairs are clustered with a radius of ±30%, with several outliers. This indicates variable performance for interanalyte precision.

The majority of the results from this laboratory demonstrated recoveries between 70-100%, relative to the design value. Three parameters were over-recovered in the high spike (Sample EXTC), Fluoranthene, Pyrene, and Bis(2-ethylhexyl)phthalate. This may be due to random laboratory contamination, as the results for Sample EXTB were not affected. One parameter, Benzylbutylphthalate, appears to have been difficult to recover by this laboratory. In Sample EXTB the recovery was 8% and in Sample EXTC the recovery was 7%. Phthalates are common laboratory contaminants. This laboratory may have had a high laboratory blank result for this parameter and by subtracting that value from the day's results, over-corrected the interlaboratory study results. Participants were not required to submit their daily QC values with the interlaboratory study results. The centre of the cluster of data pairs (Figure 11) is approximately 75-80%, indicating a slight low bias.

There is no pattern in the results across the scan for Sample EXTB. However the results for Sample EXTC have higher recoveries for the parameters eluting at the end of the scan (Figure 5). The lower recoveries of the parameters eluting at the beginning of the scan may be due to a higher temperature setting on the gas chromatogram, causing some volatilization of these compounds.

The problem parameters noted above differed from the interlaboratory mean and median by more than the standard deviation. Most of the other results are within one standard deviation of the interlaboratory mean. However the results for Benzo(g,h,i)perylene and Benzo(k)fluoranthene in Sample EXTB are higher than the interlaboratory mean and median by more than one standard deviation.

LABORATORY 2004

The results from this laboratory demonstrate some problems regarding within-laboratory interanalyte repeatability (Figure 12). Most data pairs are outside the precision limits. The clustering of the data points above the upper precision limit indicates problems with Sample EXTC, discussed in detail below. The one outlier to the right suggests contamination of Sample EXTB for the one parameter.

The interanalyte variability is satisfactory, despite the problems with sample EXTC. The majority of data pairs are clustered within a radius of ±20%.

This laboratory's results are variable relative to the design value. In Sample EXTB, Benzylbutylphthalate was not detected but in Sample EXTC a result giving 122% recovery was reported. In Sample EXTB Bis(2-ethylhexyl)phthalate was over-recovered but in Sample EXTC a result giving 101% recovery was reported. The accompanying blank sample, EXTA, was contaminated with Bis(2-ethylhexyl)phthalate, and this compound appears to have contaminated

Sample EXTB. There is no clear explanation as to why Sample EXTC was not also contaminated. In Sample EXTB a low result was reported for Di-n-Butyl phthalate, giving a recovery of 32%, but in Sample EXTC the result gave a recovery of 82%. The majority of results for Sample EXTB have recoveries between 60-85%, relative to the design value. The majority of results for Sample EXTC have recoveries between 100-125%, relative to the design value.

The centre of the cluster of data pairs (Figure 12) differs for the two samples. For Sample EXTB the results centre at approximately 805, suggesting a slight low bias. For Sample EXTC the results cluster around 115%, suggesting a slight high bias. There may be several different sources for this discrepancy between the two samples. There may be a slope problem with the calibration curve, or the preparation method may be less sensitive for the lower level sample.

The results across the scan do not demonstrate any pattern (Figures 2 and 6). Problem parameters were mentioned above.

Most of the results reported for Sample EXTB are within one standard deviation of the inter-laboratory mean, except for Dibenzo(a,h)anthracene, 2,6-Dinitrotoluene and the problem parameters noted above. However many of the results for Sample EXTC are biased high relative to the interlaboratory mean and median. Results for Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, 2-Chloronaphthalene, Chrysene, Dibenzo(a,h)anthracene, Bis(2-chloroethyl)ether, 2,4-Dinitrotoluene, 2,6-Dinitro-toluene, and Bis(2-chloroethoxy)methane are all higher than the interlaboratory mean and median by more than one standard deviation.

LABORATORY 2005

The results demonstrate problems with regard to within-laboratory interanalyte repeatability (Figure 13). One group of parameters are within the precision limits, but many of the paired data points are outside the limits.

There is considerable spread along the target line, indicating systematic bias across the scan. The spread of results is >30%, demonstrating very variable interanalyte performance.

This laboratory's results were variable relative to the design value. They did not detect Anthracene, Benzo(a)anthracene, Benzo(a)pyrene, and Benzo(g,h,i)perylene in Sample EXTB. They did not detect Benzo(a)pyrene and Benzo(g,h,i)perylene in Sample EXTC. They had a low result for Acenaphthene in Sample EXTB and low results for Pyrene in both samples. These parameters are all polyaromatic hydrocarbons (PAH's). The results suggest that the extraction procedure used by this laboratory is not effective for the analysis of this group of compounds.

The data pairs do not demonstrate a central tendency (Figure 13). A small group of parameters demonstrates satisfactory interanalyte recovery (centred around 90%). The spread of the remaining data pairs indicates low to very low bias, particularly for the high spike (Sample EXTC). These reflect the problems noted above for the PAH's.

The results across the scan do not demonstrate any pattern (Figures 2 and 6). The results are variable based on the problem parameters noted above. This is evident for both the high and low spikes.

This laboratory's results were variable relative to the interlaboratory mean and median. The problem parameters noted above were more than one standard deviation lower than the interlaboratory mean and median. In Sample EXTB the result for 4-Bromophenyl phenyl ether was greater than the interlaboratory mean and median by more than one standard deviation. In Sample EXTC the results for Fluorene, Benzylbutylphthalate, Di-n-butylphthalate, and 2,4-Dinitrotoluene were higher than the interlaboratory mean and median by more than one standard deviation.

LABORATORY 2006

The results demonstrate satisfactory within-laboratory interanalyte repeatability for almost all of the parameters (Figure 14). The interanalyte variability is satisfactory, with the data pair clustered within a radius of ±20%. Four parameters are just outside the precision limits.

The results from this laboratory were biased low relative to the design value. Recoveries ranged from 30-50% in both samples, with only one parameter, Bis(2-ethylhexyl)phthalate in Sample EXTC having a recovery of 78%. This parameter was detected in the blank sample (EXTA). This is a common laboratory contaminant, but there is no explanation as to why only one of the samples was contaminated. One low result was observed for 2,4-Dinitrotoluene in Sample EXTB. The centre of the cluster of data pairs is less than 50%, indicating a very low bias relative to the design value.

The results across the scan do not display any pattern (Figures 2 and 6). Except for the two problem parameters noted above, this laboratory had consistent results across the scan.

This laboratory's results were biased low relative to the interlaboratory mean and median. More than 60% of their results were less than the interlaboratory mean by more than a standard deviation. The combination of being biased low relative to the design values and to the interlaboratory mean and median, suggests that the calibration standard of this laboratory is inaccurate. This laboratory's standard appears to be too concentrated, resulting in the low bias for the sample results.

LABORATORY 2007

This laboratory did not report results for Bis(2-Chloroethyl)ether and Bis(2-Chloroethoxy)methane.

The results demonstrate a bias regarding the within-laboratory interanalyte repeatability (Figure 15). The majority of data points are grouped together, but are shifted away from the target precision line by the under-recovery of sample EXTC. This problem may be due to the laboratory's method extraction efficiency noted below. The interanalyte variability was satisfactory for most parameters. The results are clustered within a radius of ±20%.

The results for the reported parameters were biased low relative to the design value. This was more pronounced for Sample EXTC (High spike), which had most recoveries ranging from 40-50%. Most of the results for Sample EXTB were between 60-70%. As a result, the cluster of results for EXTB are centred around 70% (Figure 15), demonstrating a low bias. The results for EXTC are centred around 35%, demonstrating a very low bias.

The results across the scan do not display any pattern (Figures 3 and 7). This laboratory had consistent results across the scan.

Many of this laboratory's results were biased low relative to the interlaboratory mean and median. This was more pronounced for Sample EXTC. The combination of being biased low relative to the design values and to the interlaboratory mean and median, suggests that the calibration standard of this laboratory is inaccurate. This laboratory's standard appears to be too concentrated, resulting in the low bias for the sample results.

The lower recovery of the high spike (Sample EXTC) compared to the low spike also suggests that this laboratory's extraction procedure may not be as efficient when higher levels of the target parameters are present.

LABORATORY 2008

Laboratory 2008 added a note to qualify all of their data. A technical error occurred during the extraction of the samples, with excess derivatizing reagent being added to the samples. The chromatography was significantly affected, with both peak shape and retention times altered. There was insufficient sample to repeat the extraction.

This laboratory reported a combined result for Benzo(b)fluoranthene and Benzo(k)fluoranthene. The total result was evenly divided between the two isomers for the purposes of statistical calculations.

The results from this laboratory were included in the tables in Appendix 1 and plotted in Figures 3, 7, and 16. However, the technical error noted above resulted in very variable data that is not possible to assess.

LABORATORY 2010

This laboratory quantitated their result for Benzo(k)fluoranthene based on their standard for Benzo(b)fluoranthene.

The results demonstrate variable within-laboratory interanalyte repeatability (Figure 17). Most paired data points are within the precision limits. Several points are outside the limits, reflecting several problem parameters discussed below. The majority of data pairs are within a radius of ±30%, indicating variable interanalyte performance. However several parameters have very variable performance.

The results demonstrated good recovery relative to the design value for most parameters. A low result was reported for 2,4-Dinitrotoluene in Sample EXTB. Benzylbutylphthalate was under-recovered in both samples. Fluoranthene and Pyrene were over-recovered in Sample EXTC. However most results ranged from 80-120% recovery. The main cluster of data pairs is centred at approximately 110%, demonstrating satisfactory interanalyte recovery. However there are some parameters that demonstrate a high bias, particularly for Sample EXTC. There are also a few parameters that demonstrate a very low bias for Sample EXTB.

A slight pattern of increased recovery of parameters eluting in the middle third of the scan is demonstrated by both samples (Figures 3 and 7). Since temperature programs for the gas chromatograph are consistent at this point, and the pattern does not continue through to the end of the scan, this variability is probably due to a difference in the calibration standard used by this laboratory compared to the spiking material.

This laboratory's results were biased slightly high compared to the interlaboratory mean and median. While many parameters were within one standard deviation, more parameters were within two standard deviations of the interlaboratory mean and median. However this laboratory demonstrated better agreement with the design values than did most other participants.

LABORATORY 2011

The results demonstrated consistent within-laboratory interanalyte repeatability (Figure 18). Only two data points were just outside the lower precision limit. The unusual groupings of the data pairs is due to the rounding of results as reported by this laboratory. If the results included one more significant figure, the plot would have a more typical scatter of the results. The results are approximately grouped in a radius of ±20%, demonstrating satisfactory interanalyte variability.

This laboratory demonstrated good recovery relative to the design values. Most results were between 70-100%, with four results greater than 100%, but less than 120%. 2,4-Dinitrotoluene was under-recovered in Sample EXTB (33%) and Fluorene was under-recovered in sample EXTC (48%).

The unusual grouping of results noted above makes it difficult to determine the central grouping of the results. The centre of the data appears to be approximately 95% for Sample EXTB (Figure 18), indicating satisfactory interanalyte recovery for the low spike. The centre of the data for Sample EXTC appears to be approximately 80%, indicating a slight low bias for the high spike.

The results across the scan do not display any pattern (Figures 4 and 8). This laboratory had consistent results across the scan except for 2,4-Dinitrotoluene as noted above.

All of this laboratory's results were within one standard deviation of the interlaboratory mean and median. Many of the results were on the high side of the mean and median, except for 2,4-Dinitrotoluene. The results demonstrate closer agreement with the design values for most parameters than that achieved by most of the other participants.

LABORATORY 2012

The results from this laboratory demonstrate very good precision for many of the parameters but variable precision for several others (Figure 19). While most data pairs are within the precision limits, the data has considerable spread along the line for the low spike, Sample EXTB. The data are clustered within a radius of ±20% for the high spike (Sample EXTC), demonstrating satisfactory interanalyte variability. However the radius of result for Sample EXTB (low spike) is ±30%, indicating variable interanalyte performance. There is one extreme outlier discussed below.

The results demonstrated a constant level of recovery relative to the design value for most parameters. The percent recovery for most parameters was between 50-80% (Table 2, Appendix 1). The data pairs (Figure 19) are centred around approximately 60% for both samples, indicating a low bias. These results suggest that the calibration standard for this laboratory is more concentrated than the design value, resulting in the low bias.

Bis(2-ethylhexyl)phthalate was over-recovered in both EXTB and EXTC. A value of 3.1 ppb was reported in the blank (Sample EXTA). Laboratory 2012 included the results for their own lab blank with the interlaboratory study results. It had a level of 9.5 ppb for Bis(2-ethylhexyl)phthalate. This is a common laboratory contaminant as phthalates are used as plasticizers. This laboratory appears to be aware of its problem with this contaminant.

The results across the scan demonstrated a pattern of decreasing recovery for the parameters eluting at the end of the scan for Sample EXTB (Figure 4). The same pattern was not demonstrated for Sample EXTC. In Sample EXTC (Figure 8), the parameters eluting at the beginning of the scan demonstrated a pattern of increasing recovery. There is no obvious explanation as to why there would be two different patterns for the two different spiking levels. This difference in pattern contributes to the greater spread in the precision of results for EXTB noted above and demonstrated in Figure 19.

Almost all of this laboratory's results were within one standard deviation of the interlaboratory mean and median. The results for Bis(2-ethylhexyl)phthalate were greater than the mean and median due to the contamination noted above. The results for Naphthalene and Bis(2-chloroethyl)ether were lower than the interlaboratory mean and median by more than one standard deviation, but were within two standard deviations.

LABORATORY 2013

The results from this laboratory demonstrate consistent within-laboratory interanalyte repeatability (Figure 20) for most parameters. Several parameters were just outside the upper precision limits. The one extreme point reflects the problems with the results for Fluoranthene discussed below. Most of the data points are clustered within a radius of ±20%, indicating satisfactory interanalyte variability.

The results demonstrated a constant level of recovery relative to the design value for most parameters. Recovery ranged from 70-110% for most parameters. Three parameters appeared to be more difficult to analyze by the laboratory. 2,6-Dinitrotoluene and Fluorene were under-recovered in both Samples EXTB and EXTC. Fluoranthene was under-recovered in Sample EXTB (20%) but was over-recovered in Sample EXTC (143%). A small amount of Bis(2-ethylhexyl)phthalate and Di-n-butylphthalate were reported in the blank sample (EXTA). While the low spike (EXTB) may also have been contaminated with these two compounds, this effect did not occur in the high spike (EXTC). Phthalates are common laboratory contaminants due to their use as plasticizers.

The cluster of the data pairs is centred at 85% for Sample EXTB (low spike). This indicates a slight low bias (Figure 20). However the cluster of data pairs is centred at 90% for the high spike (EXTC), indicating satisfactory interanalyte recovery. This suggest that this laboratory's method is not quite as effective for low-level samples.

The results across the scan do not demonstrate any patterns (Figures 4 and 8). This laboratory

had consistent results across the scan except for the problem parameters noted above.

Most of this laboratory's results were within one standard deviation of the interlaboratory mean and median, except for Fluorene and Fluoranthene in Sample EXTB, and Fluoranthene and 2,4-Dinitrotoluene in Sample EXTC.

5.2 Acid Extractables (MISA Group 20)

OVERVIEW OF INTERLABORATORY PERFORMANCE

The graphical representation of the results arranged in elution order (Figures 26-22) indicate that the parameters in this scan are more difficult to analyze. The various laboratories demonstrate variable performance, with some labs performing consistently across the scan, while others demonstrate patterns of increasing or decreasing recovery, based on gas chromatograph elution order. With only two samples from each participant, it is not possible to determine if the laboratory's extraction procedure or the temperature program of the gas chromatograph caused the varying pattern of recoveries of the parameters across the scan.

The interlaboratory mean for all parameters, except for 4-Chloro-3-methylphenol in EXTB, and 4,6-Dinitro-o-Cresol and Pentachlorophenol in EXTC, was lower than the design value. For Sample EXTB the interlaboratory mean was 0.68-4.13 ppb lower than the design value (11-69% lower). For Sample EXTC the interlaboratory mean was 0.04-9.43 ppb lower than the design value (0.2-45% lower).

The interlaboratory median was lower than the design value for all parameters in both samples. For Sample EXTB the interlaboratory median was 1.0-4.7 ppb lower than the design value (17-78% lower). For Sample EXTC the interlaboratory median was 0.7-13.15 ppb lower than the design value (3-63% lower).

Some of the participants exhibited a low bias relative to the design value and to the interlaboratory mean and median. This was particularly noticeable for Laboratory 2006 and 2007. However, some of the participants had improved performance in the high spike (EXTC) as compared to the low spike (EXTB). This suggests that the extraction procedures used by these laboratories are not as efficient for higher level samples.

None of the participants demonstrated an overall high bias relative to the design values, except for some individual parameters. There were no false positives or evidence of laboratory contamination in the unspiked sample (EXTA).

Some individual parameters were over- or under-recovered by different participants, but there were no trends for specific parameters. There was more variability in the results for the MISA Group 20 scan than for the other two scans in this interlaboratory study. This increased variability makes it difficult to determine if the differences among the laboratories are due to differences in standards or differences in analytical methods.

The Youden two-sample plots for each individual laboratory (Figures 27-38) demonstrate that approximately 60% of the laboratories are able to maintain within-laboratory interanalyte variability of ±20%. Individual parameters may be a problem for an individual laboratory. Most of the graphs demonstrate a systemic bias across the scan as evidenced by the spread of the data pairs along the line of target precision.

Due to the large number of parameters included in MISA Group 20, this report does not attempt to assess the between-laboratory performance of individual parameters. With only two spiked

samples analyzed by each participant, there is insufficient data to assess the analytical characteristics of each parameter. Future studies will attempt to assess the analytical characteristics of individual parameters by submitting more samples to the participants and including duplicate analyses.

The following sections review individual laboratory performance. Performance was assessed according to within-laboratory interanalyte repeatability), percent recovery relative to the design value (interanalyte bias), performance across the scan, and percent recovery relative to the interlaboratory mean and median.

LABORATORY 2001

The samples for this scan were analyzed using GC/MS. This laboratory did not detect 2,3,5-Trichlorophenol in both of the spiked samples. However they did report detecting 2,3,4-Trichlorophenol in the correct range that was spiked in the samples. These two isomers elute close together from the gas chromatograph, and this laboratory may have identified the wrong isomer. The spiking level of 4,6-Dinitro-o-Cresol in both samples was below the detection limit of this laboratory. The spiking level of 2,4-Dimethylphenol in Sample EXTB was also below the detection limit of this laboratory.

The results from this laboratory demonstrate satisfactory within-laboratory interanalyte repeatability (Figure 27). The majority of the paired results are within the precision limits, with only two results just outside the precision limits. However the interanalyte performance is very variable as the data are spread across an area with a radius of greater than ±30%.

The results from this laboratory demonstrated consistent recovery relative to the design value for most parameters, except for specific parameters noted above. The percent recovery for most parameters was between 80-120% (Table 2, Appendix 1). However there is no easily distinguished cluster of results, so it is difficult to assess a ranking for interanalyte recovery. Some parameters have a slight low bias and others have a slight high bias.

The performance across the first half of the scan is consistent for both the low and high spikes (Figures 21 and 24, Appendix 1). A slight decrease in recovery for the parameters eluting during the final third of the scan is noted for both samples.

Laboratory 2001's results were generally close to, or higher, than the interlaboratory mean and median, except for 2,3,5-Trichlorophenol and 4,5-Dinitro-o-Cresol, as noted above. Several parameters in both samples differed from the interlaboratory mean by more than one standard deviation (Table 1, Appendix 1). However, the results were within one standard deviation of the target value.

LABORATORY 2002

This laboratory reported a combined result for 2,3,4,5-Tetrachlorophenol, 2,3,4,6-Tetrachlorophenol, and 2,3,5,6-Tetrachlorophenol. The total result was evenly divided between the three isomers for the purposes of statistical calculations. They also did not detect 2,3,5-Trichlorophenol in the two spiked samples. However they did report detecting

2,3,4-Trichlorophenol in the correct range that was spiked in the samples. These two isomers elute close together from the gas chromatograph, and this laboratory may have identified the wrong isomer.

The results demonstrated consistent within-laboratory interanalyte repeatability (Figure 28). The majority of results are close to the line of target precision. The one outlying point reflects a problem parameter (2,4-Dichlorophenol) discussed below. There are two groups of parameters evident in Figure 28. One group displays excellent interanalyte precision, with a the cluster of results having a radius of ±10%. The second group of parameters is clustered with a radius of ±20%, demonstrating satisfactory interanalyte precision.

This laboratory demonstrated a constant level of recovery relative to the design value for most parameters. The majority of parameters had recoveries between 50-80% for both spiked samples (Table 2, Appendix 1). One parameter was over-recovered in Sample EXTB, 2,4-Dichlorophenol. This may be due to random unidentified laboratory contamination, as the results for Sample EXTC were not affected. The results group into two clusters as noted above. One cluster is centred on 80% and demonstrates a slight low bias. The other cluster is centred on approximately 50%, demonstrating a low bias. The low bias relative to the design value may be due to a difference in standard between this laboratory and the spiking material.

The results across the scan are consistent and do not demonstrate any patterns (Figures 21 and 24). Problem parameters were noted above.

The problem parameter noted above differed from the interlaboratory mean by more than the standard deviation. The other results were within one standard deviation of the interlaboratory mean and median.

LABORATORY 2003

This laboratory did not detect 2,3,5-Trichlorophenol in both spiked samples.

This laboratory had fairly consistent within-laboratory interanalyte repeatability (Figure 29). Only two parameters were outside the precision limits of ±20%. However there is considerable variation in interanalyte precision. The results are spread in an area with a radius greater than ±30%, demonstrating very variable performance.

The majority of the results from this laboratory demonstrated recoveries between 40-60%, relative to the design value. Pentachlorophenol was slightly over-recovered in the high spike (Sample EXTC), relative to the design value. This may be due to random laboratory contamination, as the results for Sample EXTB were not affected. 4-Nitrophenol appeared to be more a difficult parameter for this laboratory to analyze, as the recoveries in samples EXTB and EXTC were 28% and 36%, respectively. The results are centred at approximately 50%, demonstrating a low bias low relative to the design value. This may be due to a difference in standards between this laboratory and the spiking material.

There was no pattern in the results across the scan for both samples (Figures 21 and 24).

Most of the results from this laboratory were low relative to the interlaboratory mean and median. Most of the results were within one standard deviation of the mean. The results appeared more

variable compared to the interlaboratory median, due to the variability of the interlaboratory median compared to the target value.

LABORATORY 2004

This laboratory reported a combined result for 2,3,4,5-Tetrachlorophenol and 2,3,4,6-Tetrachlorophenol. The total result was divided between the two isomers for statistical purposes.

The results from this laboratory demonstrates problems regarding within-laboratory interanalyte repeatability (Figure 30). All the data pairs are outside the precision limits. This laboratory's results are very variable. The spread of the data points above the upper precision limit indicates problems with Sample EXTB (low spike), discussed below.

This laboratory did not detect Phenol, 4-Nitrophenol, and 4,6-Dinitro-o-Cresol in Sample EXTB (low spike). In Sample EXTB 2,3,5-Trichlorophenol was under-recovered (15%), but in Sample EXTC a result giving 140% recovery was reported. A similar effect was noted for 2,4,6-Trichlorophenol. These results suggest that the extraction method used by this laboratory is not as sensitive for low-level samples. Their overall results were variable relative to the design value. This laboratory did note in their report that they did not correct for percent recovery for 4-Nitrophenol.

There is no clustering of the data pairs from this laboratory (Figure 30), therefore it is not possible to rank the interanalyte variability or to rank the interanalyte recovery.

The results across the scan do not demonstrate any pattern (Figures 21 and 24), dispite the precision problems noted above. Individual problem parameters were mentioned above.

The results are variable compared to the interlaboratory mean and median. Some parameters are higher than the mean and median, and some parameters are lower. Many of the results differ by more than one standard deviation from the mean or the median. This reflects the very variable performance of this laboratory.

LABORATORY 2005

The results from this laboratory demonstrate problems with regard to within-laboratory interanalyte repeatability (Figure 31). Some of parameters are within the precision limits, but many of the paired data points are outside the limits, on the high side. The low recovery from Sample EXTB causing the shift for may of the data pairs, suggests that this laboratory's method is less sensitive to low-level samples. This laboratory's interanalyte performance is very variable.

This laboratory's results were variable relative to the design value, with a tendency to be biased low. This was more noticeable in the low spike (Sample EXTB) than the high spike (Sample EXTC). Laboratory 2005 did not report any results for p-Cresol. They did not detect 2,3,5-Trichlorophenol in either of the spiked samples. However they did report detecting 2,3,4-Trichlorophenol in the correct range that was spiked in the samples. These two isomers elute close together from the gas chromatograph, and this laboratory may have identified the wrong isomer. They over-recovered 2,3,4,5-Tetrachlorophenol, 2,3,4,6-Tetrachlorophenol, and 2,3,4,5-Tetrachlorophenol in Sample EXTC (high spike). However the data pairs do not form a cluster and therefore it is not possible to assign a rank for interanalyte recovery.

The results demonstrate a slight pattern of increasing recovery across the scan, except for 4,6-Dinitro-o-Cresol and Pentachlorophenol. This pattern is more marked in the high spike (EXTC, Figure 25) than the low spike (EXTB, Figure 22).

This laboratory's results were variable relative to the interlaboratory mean and median, with some parameters higher and some parameters lower. However, all the results were within one standard deviation of the interlaboratory mean and median, except for the three Tetrachlorophenols noted above for Sample EXTC.

LABORATORY 2006

The results from this laboratory demonstrate satisfactory within-laboratory interanalyte repeatability for most of the parameters (Figure 32) in this scan. Two parameters are just outside the upper precision limit. The interanalyte performance is satisfactory for the central group of parameters within a radius of ±20%. However there is a group of parameters that were not detected in the low spike (EXTB) that demonstrate variable performance.

The results from this laboratory were biased low relative to the design value. Recoveries ranged from 20-60% in both samples for most parameters. The spiking level of 4,6-Dinitro-o-Cresol in Sample EXTB was below this laboratory's detection limit. They did not detect 2,4-Dimethylphenol, 2-Chlorophenol, 4-Nitrophenol, and Phenol in the low spike (EXTB). 2,3,5,6-Tetrachlorophenol was over-recovered in Sample EXTC. 4,6-Dinitro-o-Cresol was not detected in the high spike (EXTC).

The data forms two groups (Figure 32). One group of data is clustered around 50%, and demonstrates a low bias. The second group of data is on the vertical axis due to non-detection in sample EXTB. This interanalyte recovery for these parameters indicates a very low bias.

The results across the scan display a slight pattern of increasing recovery across the scan. This is more evident in the high spike (Figure 25) than for the low spike (Figure 22).

This laboratory's results were biased low relative to the interlaboratory mean and median. The results for the low spike were within one standard deviation of the interlaboratory mean and median. Most of the results for the high spike differed from the interlaboratory mean and median by more than one standard deviation. The combination of being biased low relative to the design values and to the interlaboratory mean and median, suggests that the calibration standard of this laboratory is inaccurate. This laboratory's standard appears to be too concentrated, resulting in the low bias for the sample results.

LABORATORY 2007

This laboratory did not have standards available to report results for 2,4-Dimethylphenol, 2-Chlorophenol, and Phenol. They also noted that they were waiting to receive a new chlorophenol standard. This had not arrived in time for the analysis of the interlaboratory study, therefore they calibrated their instrument using an old standard. They noted that any inaccuracies in their results should be attributed to this problem.

The results demonstrate good within-laboratory interanalyte repeatability (Figure 33) for this scan. All of the data pairs are within the precision limits. The interanalyte performance is variable, as the results are within a radius of ±30%.

The results were biased low relative to the design value. This may be attributed to the old calibration standard used by this laboratory as noted above. The cluster of data pairs are centred at about 55%.

The results across the scan do not display any pattern. This laboratory had consistent results across the scan (Figures 22 and 25).

Many of this laboratory's results were biased low relative to the interlaboratory mean and median. This was more pronounced for Sample EXTC. The combination of being biased low relative to the design values and to the interlaboratory mean and median, supports the concern expressed by Laboratory 2007, that their calibration standard is inaccurate. This laboratory's standard appears to be too concentrated, resulting in the low bias for the sample results.

LABORATORY 2008

Laboratory 2008 added a note to qualify all of their data. A technicial error occurred during the extraction of the samples, with excess derivatizing reagent being added to the samples. The chromatography was significantly affected, with both peak shape and retention times altered. There was insufficient sample to repeat the extraction.

The results from this laboratory were included in the tables in Appendix 1 and plotted in Figures 22, 25, and 34. However due to the technical error noted above, the results are very variable and it is not possible to assess the performance of this laboratory.

LABORATORY 2010

This laboratory did not detect 2,3,5-Trichlorophenol in both of the spiked samples. However they did report detecting a different trichlorophenol in the correct range that was spiked in the samples. This isomer was quantitated as 2,3,5-Trichlorophenol.

The results demonstrate a problem regarding within-laboratory interanalyte repeatability (Figure 35). All of the paired data points are outside the precision limits. The results reflect a high bias in the values for Sample EXTC. There is also considerable spread along the line with no clustering of the data pairs, reflecting very variable interanalyte performance.

The results demonstrated variable recovery relative to the design value for most parameters. Except for 2,4-Dimethylphenol, all the results in the low spike (EXTB) were low, relative to the design value. In the high spike (EXTC), all the results were high relative to the design value, except for Phenol and 2,3,5,6-Tetrachlorophenol. There is no clustering of the data pairs, so it is not possible to rank the interanalyte recovery from this laboratory.

A slight pattern of increased recovery of parameters eluting in the middle third of the scan is demonstrated by both samples (Figures 23 and 26). Since temperature programs for the gas chromatograph are consistent at this point, and the pattern does not continue through to the end of the scan, this variability is probably due to a difference in the calibration standard used by this laboratory, compared to the design values.

This laboratory's results for the high spike (EXTC) were biased high compared to the interlaboratory mean and median. Except for 2,4-Dimethylphenol, 4,6-Dinitro-o-Cresol, and 4-Methyl-3-Methylphenol, all the results differ from the interlaboratory mean and median by more than one standard deviation. The results for the low spike are closer to the interlaboratory mean and median. Except for the parameters that were not detected in Sample EXTB (noted above), the results for the other parameters are within one standard deviation of the mean and median.

LABORATORY 2011

This laboratory did not have a standard for 2,3,5-Trichlorophenol. They quantitated the observed peak in the samples based on their 2,3,4-Trichlorophenol standard. Their reported result for 4-Nitrophenol was below their detection limit and only an approximate value.

The results demonstrated consistent within-laboratory interanalyte repeatability (Figure 36). Only two data points were just outside the upper precision limit. However the results demonstrate very variable interanalyte performance, as the results are spread across an area with a radius greater than ±30%.

This laboratory demonstrated variable recovery relative to the design values. 2,4-Dimethylphenol and 2,3,4,5-Tetrachlorophenol were over-recovered in both samples. 2-Chlorophenol was also over-recovered in Sample EXTB. Recoveries for the other parameters ranged from 13-95%, relative to the design values. The data pairs do not demonstrate a central cluster. Some parameters appear to have a low or very low bias, while others have a slight high bias.

The results across the scan display a slight pattern of decreasing recovery across the scan (Figures 23 and 26). This laboratory should check the gas chromatographic conditions to determine a possible source for this effect.

Most of this laboratory's results were within one standard deviation of the interlaboratory mean and median. 2,3,4,5-Tetrachlorophenol differed from the interlaboratory mean and median in both samples by more than one standard deviation. In Sample EXTC, 2,3,5,6-Tetrachlorophenol and Pentachlorophenol differed from the mean and median by more than one standard deviation.

LABORATORY 2012

This laboratory did not detect 2,3,5-Trichlorophenol in both of the spiked samples. However they did report detecting 2,3,4-Trichlorophenol in the correct range that was spiked in the samples. These two isomers elute close together from the gas chromatograph, and this laboratory may have identified the wrong isomer.

The within-laboratory interanalyte repeatability is good for most parameters (Figure 37). Three parameters are outside the precision limits. Except for the three parameters, the results demonstrate satisfactory interanalyte variability, as the results are within a radius of ±20%.

The results from this laboratory demonstrated good recovery relative to the design value for most parameters. The percent recovery for most parameters was between 70-100% (Table 2, Appendix 1). 4-Nitrophenol was under-recovered in the high spike (EXTC). 4.6-Dinitro-o-Cresol and 4-Nitrophenol were not detected in the low spike (EXTB). Pentachlorophenol was over-recovered in the high spike (EXTC). The results are clustered around 85% (Figure 37), demonstrating a slight low bias for interanalyte recovery.

The results across the scan demonstrated a consistent pattern of recovery (Figures 23 and 26), with no marked patterns.

Almost all of this laboratory's results were within one standard deviation of the interlaboratory mean and median. The result for Pentachlorophenol was greater than the mean and median by more than one standard deviation in Sample EXTB, the result is almost exactly the design value (reported = 6.1 ppb, design = 6.0 ppb). The results 4-Nitrophenol also differed from the mean and median by more than one standard deviation.

LABORATORY 2013

This laboratory did not detect 2,3,5-Trichlorophenol in both of the spiked samples. However they did report detecting 2,3,4-Trichlorophenol in the correct range that was spiked in the samples. These two isomers elute close together from the gas chromatograph, and this laboratory may have identified the wrong isomer.

The within-laboratory interanalyte repeatability is shifted outside the lower precision limits due to the lower recoveries in Sample EXTC (Figure 38). 4,6-Dinitro-o-Cresol was over-recovered by a very large amount (see below) and was excluded from Figure 38 so as not to distort the scale. The results demonstrate variable interanalyte recovery for Sample EXTB, with most of the data pairs within a a radius of ±30%. However for Sample EXTC, the data pairs are spread over an area with a radius greater than ±30%, demonstrating very variable interanalyte performance.

The results from this laboratory demonstrated variable recovery relative to the design value for most parameters. 4,6-Dinitro-o-Cresol was over-recovered by more than a factor of 10 in both samples. The results for this parameter from Laboratory 2013 were excluded from the statistical calculations due to the extreme difference in results compared to the other participants. The results for the low spike (EXTB) ranged from 27-183% recovery, with most parameters between 52-93% recovery, relative to the design value. The results for the high spike (EXTC) ranged from 3-143%, with most parameters between 20-81% recovery, relative to the design value. The generally lower recovery for the high spike suggests that the extraction efficiency of this laboratory's method is not as suitable for high level samples. The data pairs are clustered around 85% (Figure 38) for the low spike (EXTB), demonstrating a slight low bias for interanalyte recovery. However for the high spike (EXTC), the data pairs are clustered around 50%, demonstrating a low bias for interanalyte recovery.

The results across the scan demonstrate a pattern of increasing recovery (Figures 23 and 26). Some adjustments to the gas chromatogram temperature programming may be necessary by this laboratory to eliminate this pattern.

This laboratory's results were variable compared to the interlaboratory mean and median. Several parameters were within one standard deviation of the mean and median, but an equal number were higher or lower by more than one standard deviation.

5.3 Neutral Chlorinated Extractables (MISA Group 23)

OVERVIEW OF INTERLABORATORY PERFORMANCE

The graphical representation of the results arranged in elution order (Figures 39-42) indicate that the parameters in the Neutral Chlorinated scan have similar patterns of recovery as the Base/Neutral scan (MISA Group 19). Most participants demonstrated patterns of consistent recovery across the scan, though Laboratory 2006 did demonstrate a slight pattern of increasing recovery.

The interlaboratory mean for all parameters was lower than the design value. For Sample EXTB the interlaboratory mean was 1.81-2.89 ppb lower than the design value (30-48% lower). For Sample EXTC the interlaboratory mean was 6.12-10.06 ppb lower than the design value (29-48% lower).

The interlaboratory median was lower than the design value for all parameters in both samples. For Sample EXTB the interlaboratory median was 1.65-2.65 ppb lower than the design value (28-44% lower). For Sample EXTC the interlaboratory median was 4.65-11.6 ppb lower than the design value (22-55% lower).

Most of the participants exhibited a low bias relative to the design value and to the interlaboratory mean and median. However the difference from the interlaboratory mean and median was not as pronounced as the difference from the design value.

None of the participants demonstrated an overall high bias relative to the design values, except for some individual parameters. However Laboratory 2002 and 2005 were high compared to the interlaboratory mean and median, but had very good agreement with the design values. The results suggest that the differences among the laboratories is primarily due to differences in standards for the MISA Test Group.

Only one laboratory, 2001, detected any of the parameters in this scan in the unspiked sample (EXTA). The levels were very low and do not appear to be a contamination problem.

Some individual parameters were over- or under-recovered by different participants, but there were no trends for specific parameters.

The Youden two-sample plots for each individual laboratory (Figures 43-52) demonstrate that approximately 60% of the laboratories are able to maintain within-laboratory interanalyte variability of ±20%. Individual parameters may be a problem for an individual laboratory. Several of the graphs demonstrate a systemic bias across the scan as evidenced by the spread of the data pairs along the line of target precision.

Most of the parameters included in MISA Group 23 have not been included in any previous interlaboratory studies conducted by the Quality Assurance Office, LSB. Therefore this report does not attempt to assess the between-laboratory performance of individual parameters. With only two spiked samples analyzed by each participant, there is insufficient data to assess the analytical characteristics of each parameter. Future studies will attempt to assess the analytical characteristics of individual parameters by submitting more samples to the participants and including duplicate analyses.

The following sections review individual laboratory performance. Performance was assessed according to within-laboratory interanalyte repeatability, percent recovery relative to the design value (interanalyte bias), performance across the scan, and percent recovery relative to the interlaboratory mean and median.

LABORATORY 2001

The samples for this scan were analyzed using GC/ECD. There were low levels of Hexachlorobenzene, Hexachloroethane, and Hexachlorobutadiene reported in the blank sample, EXTA.

The results demonstrate some problems with within-laboratory interanalyte repeatability (Figure 43). The results are outside the upper precision limits, due to the low recovery of Sample EXTB. The interanalyte performance is very variable, as the data pairs are spread along an area greater than ±30%.

The results from this laboratory were low relative to the design value for all parameters. This was more pronounced in the low spike (EXTB) than the high spike (EXTC). They did not detect 1,2,4-Trichlorobenzene in both of the spiked samples. The percent recovery for most parameters was between 12-63% for EXTB (Table 6, Appendix 1). For Sample EXTC the recovery ranged between 33-90%. The results suggest that the extraction procedure used by this laboratory is not as effective for low-level samples. There is not a distinct clustering of the results (Figure 43), but the results are generally centred around 40%, demonstrating a very low bias for interanalyte recovery.

The performance across the scan is similar for both the low and high spikes (Figures 39 and 41). There is a slight tendency towards increasing recovery towards the end of the scan.

Laboratory 2001 demonstrated variable performance relative to the interlaboratory mean and median. Several parameters were lower than the mean and median by more than one standard deviation.

LABORATORY 2002

The results from this laboratory demonstrate satisfactory within-laboratory interanalyte repeatability (Figure 44). The majority of results are close to the line of target precision and within a radius of ±20%. Two parameters were just outside the lower precision limit.

This laboratory demonstrated good recovery relative to the design value for most parameters. The majority of parameters had recoveries between 70-110% for both spiked samples (Table 6, Appendix 1). The results are centred on 100%, demonstrating excellent interanalyte recovery.

The results across the scan are consistent and do not demonstrate any patterns (Figures 39 and 41).

This laboratory's results were biased high compared to the interlaboratory mean and median. Most of the results were greater than the interlaboratory mean and median by more than one standard deviation. However this laboratory's results were within one standard deviation of the design values for both samples.

LABORATORY 2003

This laboratory analyzed the full scan for MISA Group 23 using GC/ECD. Three parameters, Hexachlorobenzene, Hexachloroethane, and Hexachlorobutadiene, were also part of the GC/MS scan for MISA Group 19, and a second set of values was reported for these parameters. The results from the GC/ECD analysis were used for the interlaboratory statistical calculations and for the graphs, as this is the analytical techniques used by this laboratory for MISA samples. The GC/MS results are provided for information only.

The results demonstrate consistent within-laboratory interanalyte repeatability (Figure 45). All the paired results were within the precision limits. However the results are spread across an area of ±30%, indicating variable interanalyte performance.

The majority of the results from this laboratory were low relative to the design value. Recoveries ranged between 22-89%. The group of data pairs are approximately centred around 50%, indicating a low bias for interanalyte recovery.

The performance across the scan is similar for both the low and high spikes (Figures 39 and 41). The results are generally consistent across the scan and demonstrate no marked patterns.

Laboratory 2003 was biased low relative to the interlaboratory mean and median. However most parameters were within one standard deviation of the mean and median.

LABORATORY 2004

The results for Hexachlorobutadiene and Hexachloroethane in Sample EXTB were qualified with the remark "Trace". The laboratory's MDL's were not available for these parameters. The results were included with the interlaboratory statistical calculations and the graphs, but should be treated with caution.

The results from this laboratory demonstrate an unusual pattern with regard to within-laboratory interanalyte repeatability (Figure 46). Most data pairs are within the precision limits. However, there are problems with interanalyte variability, as the data points are grouped in pairs of parameters spread quite far apart. The data are spread over a range of greater than ±30%, demonstrating very variable performance.

This laboratory's results were variable relative to the design value, particularly for the high spike (EXTC). Excluding the two parameters noted above, the recoveries in Sample EXTB were between 17-65%. The recoveries in Sample EXTC were between 20-99%. It is difficult to determine if the low bias is due to a difference in standards or due to problems with the extraction procedure used by this laboratory. The results do not form a central cluster (Figure 46), so it is difficult to rank the interanalyte recovery. Three parameters demonstrate a slight low bias but the other four parameters demonstrate a very low bias.

The results across the scan are variable, but do not demonstrate any specific pattern (Figures 39 and 41).

The results for this laboratory were low compared to the interlaboratory mean and median, except for Hexachlorobenzene, Hexachlorobutadiene, and Hexachloroethane in Sample EXTC. Several of the parameters were lower than the interlaboratory mean and median by more than one standard deviation.

LABORATORY 2005

The results demonstrate good within-laboratory interanalyte repeatability (Figure 47). The interanalyte performance is satisfactory as the results are spread within an area of ±20%.

This laboratory's results were good relative to the design value. Only Hexachlorobenzene had recoveries over 100%, but they were less than 120%. Most recoveries ranged between 70-94%. The data pairs clustered around 85% (Figure 47) indicating a slight low bias relative to the design value.

The results across the scan demonstrates a slight pattern of increasing recovery (Figures 39 and 41). Some adjustments to the gas chromatograph temperature program may eliminate this problem.

This laboratory's results were consistent relative to the interlaboratory mean and median. Only Hexachlorobenzene differed by more than one standard deviation than the interlaboratory mean and median.

LABORATORY 2006

The results demonstrate good within-laboratory interanalyte repeatability (Figure 48), as all the data pairs are within the precision limits. However the data pairs are spread over an area greater than ±30%, indicating very variable interanalyte performance.

The results from this laboratory were biased low relative to the design value. Recoveries ranged from 16-55% in both samples, with only one parameter, 1,2,4,5-Tetrachlorobenzene, having a recovery of 80% in both samples. There is no central cluster of data pairs, so it is not possible to rank the interanalyte recovery, though all the results demonstrate varying degrees of low bias.

The results across the scan do not display any pattern (Figures 39 and 41). Except for 1,2,4,5-Tetrachlorobenzene noted above, this laboratory had consistent results across the scan.

This laboratory's results were biased low relative to the interlaboratory mean and median, except for 1,2,3,4-Tetrachlorobenzene and 1,2,4,5-Tetrachlorobenzene. The combination of being biased low relative to the design values and to the interlaboratory mean and median, suggests that the calibration standard of this laboratory is inaccurate. This laboratory's standard appears to be too concentrated, resulting in the low bias for the sample results.

LABORATORY 2007

The results from this laboratory demonstrate a bias regarding the within-laboratory interanalyte repeatability (Figure 49). The majority of data points are grouped together within a range of ±10%. This appear to indicate excellent interanalyte variability, but the cluster of results are shifted away from the target precision line by the under-recovery of sample EXTC. This problem may be due to the laboratory's method extraction efficiency discussed below.

This laboratory's results are biased low relative to the design value. This was more pronounced for Sample EXTC (High Spike), which had most recoveries ranging from 40-50%. Most of the results for Sample EXTB were between 60-75%. The cluster of data points are centred around 75% for EXTB (Figure 49), which indicates a slight low bias for this sample. However the data points are centred around 40% for EXTC (High Spike), indicating a very low bias.

The results across the scan do not display any marked pattern (Figures 40 and 42). This laboratory had consistent results across the scan, though at different levels of recovery for the two samples.

This laboratory's results have very good agreement with the interlaboratory mean and median for the low spike (EXTB). However, their results were biased low relative to the interlaboratory mean and median for the high spike (EXTC).
The lower recovery of the high spike (Sample EXTC) compared to the low spike (Sample EXTB) suggests that this laboratory's extraction procedure may not be as efficient when higher levels of the target parameters are present.

LABORATORY 2008

Laboratory 2008 added a note to qualify all of their data. A technicial error occurred during the extraction of the samples, with excess derivatizing reagent being added to the samples. The chromatography was significantly affected, with both peak shape and retention times altered. There was insufficient sample to repeat the extraction.

This laboratory also noted that there had been an error when the samples had been logged into their sample tracking system. A separate analysis is required for MISA Group 23, which they were unable to perform for this interlaboratory study, due to sample size limitations.

Due to the problems noted above, it is not possible to assess the performance for this laboratory. The results were included in the tables in Appendix 1 and in Figures 40 and 42, but the two results do not give a proper indication of this laboratory's performance. A Youden paired-sample plot was not prepared for the results from this scan.

LABORATORY 2010

The results demonstrate good within-laboratory interanalyte repeatability (Figure 50). All of the paired data points are within the precision limits. The one point that is separated from the central group of results reflects the higher recovery of Hexachlorobenzene noted below. The central group of results is clustered within a radius of ±10%, demonstrating excellent interanalyte variability.

This laboratory demonstrated consistent recovery relative to the design value for all parameters, except Hexachlorobenzene, which had a recovery of 110% in both samples. Most results ranged from 65-85% recovery. The results are centred around 75% (Figure 50), indicating a slight low bias.

The results demonstrated consistent recovery across the scan (Figures 40 and 42), except for Hexachlorobenzene, as noted above. There were no patterns to the results.

This laboratory's results were within one standard deviation of the interlaboratory mean and median. Only the results for Hexachlorobenzene were higher than the interlaboratory mean and median by more than one standard deviation.

LABORATORY 2011

The initial table of results from this interlaboratory study was distributed to the participants on September 15, 1989. Laboratory 2011 was concerned about their low results for this scan and cross-checked their results. They determined that there had been a mix-up with the sample extracts during instrumental analysis, and therefore had reported the wrong results for this interlaboratory study. They requested that their results for this scan be withdrawn. The original results reported by this laboratory are included in Table 5, Appendix 1, but were not included in the statistical calculations. Graphs were not prepared of this laboratory's results for this scan.

LABORATORY 2012

The within-laboratory interanalyte repeatability is variable due to the two different patterns across the scan discussed below. In Figure 51, there is a group of parameters demonstrating good precision. Several other parameters are outside the lower precision limit due to the low recoveries in Sample EXTC. The interanalyte variability is satisfactory for Sample EXTB (range of results within ±20%), but is variable for Sample EXTC (range of results within ±30%).

The results from this laboratory demonstrated consistent recovery relative to the design value for most parameters. The percent recovery for most parameters was between 50-80% (Table 6). Hexachloroethane and Hexachlorobutadiene had low recoveries in Sample EXTC (23% and 35% respectively). The results are centred around 75% for Sample EXTB and around 55% for Sample EXTC, indicating a low bias. These results suggest that the calibration standard for this laboratory is more concentrated than the design value, resulting in the low bias. The generally lower recoveries for Sample EXTC also suggest that the extraction procedure used by this laboratory may not be as effective for higher level samples.

The results across the scan are consistent for Sample EXTB (Figure 40). The same pattern was not demonstrated for Sample EXTC. In Sample EXTC (Figure 42), there is a pattern of increasing recovery across the scan. There is no obvious explanation as to why there would be two different patterns for the two different spiking levels.

All of this laboratory's results were within one standard deviation of the interlaboratory mean and median, except for Hexachloroethane in Sample EXTC. There is no tendency to be biased high or low.

LABORATORY 2013

The results demonstrate variable within-laboratory interanalyte repeatability (Figure 52). Several parameters were outside the upper precision limits. The results demonstrate very variable interanalyte performance, as the results are spread across an area greater than ±30%.

The results from this laboratory demonstrated variable recovery relative to the design value for most parameters. Recovery ranged from 33-195% in both samples. Hexachloroethane was over-recovered in both samples. There is no central cluster of data pairs for this laboratory (Figure 52), so it is not possible to rank the interanalyte recovery.

The results across the scan demonstrated a similar pattern for both samples (Figures 40 and 42). The first three parameters had higher recoveries, while the last four parameters dropped to much lower recoveries. This laboratory may wish to check their temperature programming of the gas chromatograph to determine if there is a sudden change taking place at the time that the latter parameters begin eluting from the column.

The results for the low spike (EXTB) were within one standard deviation of the interlaboratory mean and median, except for Hexachloroethane. In Sample EXTC, the results for Hexachloroethane, 1,23-Trichlorobenzene, and 1,2,4-Trichlorobenzene were greater than the interlaboratory mean and median by more than one standard deviation.

6 REFERENCES

1. ONTARIO REGULATION 695/88 as amended to Ontario Regulation 533/89 under the Environmental Protection Act; Effluent Monitoring - General

2. Youden, W.J. and Steiner, E.H.; 1975; Statistical Manual of the Association of Official Analytical Chemists; Association of Official Analytical Chemists; ISBN 0-935584-15-3

3. Interlaboratory Study 88-1, Organic Parameters in Reagent Water and Effluents; July 1989; Environment Ontario, Laboratory Services Branch; ISBN 0-7729-5756-8

7 APPENDIX 1 - FULL DATA SET

TABLE 1 - INTERLABORATORY STUDY 89-5
BASE/NEUTRAL EXTRACTABLE ORGANICS (MISA GROUP 19)
RESULTS EXPRESSED IN ppb

DISTRIBUTED: MAY 2, 1989

SAMPLE PARAMETERS		DESIGN (ppb)	LABORATORY NUMBER 2001	2002	2003	2004	2005	2006	2007	2008	2010	2011	2012	2013	MEAN	MEDIAN	RANGE MIN	RANGE MAX	STD DEV
EXTA	Acenaphthene	0																	
EXTA	Anthracene	0																	
EXTA	Benzo(a)anthracene	0																	
EXTA	Benzo(a)pyrene	0																	
EXTA	Benzo(b)fluoranthene	0																	
EXTA	Benzo(g,h,i)perylene	0																	
EXTA	Benzo(k)fluoranthene	0																	
EXTA	2-Chloronaphthalene	0																	
EXTA	Chrysene	0																	
EXTA	Dibenzo(a,h)anthracene	0																	
EXTA	Fluoranthene	0																	
EXTA	Fluorene	0																	
EXTA	Naphthalene	0																	
EXTA	Phenanthrene	0																	
EXTA	Pyrene	0																	
EXTA	Benzylbutylphthalate	0																	
EXTA	Bis(2-ethylhexylphthalate)	0				15		43.59					3.1	1.7					
EXTA	Di-n-butylphthalate	0						< 0.7		0.2				1.2					
EXTA	4-Bromophenyl phenyl ether	0																	
EXTA	4-chlorophenyl phenyl ether	0																	
EXTA	Bis(2-chloroethyl)ether	0																	
EXTA	2,4-Dinitrotoluene	0																	
EXTA	2,6-Dinitrotoluene	0																	
EXTA	Bis(2-chloroethoxy)methane	0																	
EXTA	N-Nitrosodi-n-propylamine	0																	
EXTB	Acenaphthene	6	4.3	5	4.7	3.7	1.05	2.19	3.8	10	6.25	5	4.8	4.3	4.59	4.5	1.05	10	2.180
EXTB	Anthracene	6	3.7	4	3.6	3.6	< DL	1.86	3.8	3	6.61	5	5	4	4.02	3.8	1.86	6.61	1.218
EXTB	Benzo(a)anthracene	6	3.4	4.4	4.3	4.4	< DL	1.43	4.4	2.1	6.12	6	3.5	5	4.10	4.4	1.43	6.12	1.442
EXTB	Benzo(a)pyrene	6	3.2	3.3	4.3	4.6	< DL	1.41	4.4	0	4.27	5	2.7	4	3.38	4	0	5	1.516
EXTB	Benzo(b)fluoranthene	6	3.7	4.8 *	4.7	4.8	3.56	1.87	4.4	1.05 *	5.2	7	2.9	5.6	4.13	4.55	1.05	7	1.636
EXTB	Benzo(g,h,i)perylene	6	3	5.1	5.4	5.4	< DL	1.78	4	0	2.87	4	2.3	5.4	3.57	4	0	5.4	1.764
EXTB	Benzo(k)fluoranthene	6	2.8	4.8 *	6.1	5.2	1.38	1.58	4.4	1.05 *	5.06 **	5	2.1	5.3	3.73	4.6	1.05	6.1	1.811
EXTB	2-Chloronaphthalene	6	0	4.9	3.6	4.4	4.3	2.1	3.6	11.2	5.98	6	4	4.1	4.52	4.2	0	11.2	2.656
EXTB	Chrysene	6	2.7	5.7	5.4	4.7	3.01	1.57	4.4	4.4	5.56	5	2.5	5.1	4.17	4.55	1.57	5.7	1.375
EXTB	Dibenzo(a,h)anthracene	6	3	4.3	4.5	5.3	2.91	1.45	4	0	1.42	4	2.4	5.3	3.22	3.5	0	5.3	1.658
EXTB	Fluoranthene	6	4.3	5	5.9	5	5.26	2.74	4.4	3	8.2	5	4.8	1.2	4.57	4.9	1.2	8.2	1.740
EXTB	Fluorene	6	4.3	5.7	4.7	5.4	5.46	2.29	4	0	6.21	4	4.5	2.3	4.07	4.4	0	6.21	1.770
EXTB	Naphthalene	6	4.5	8.7	4.2	4	5.07	1.94	3.3	5.2	5.72	7	4.2	4.4	4.85	4.45	1.94	8.7	1.736
EXTB	Phenanthrene	6	4.1	4.8	4.4	4.4	5.06	2.78	4.4	2.4	7.05	5	4.9	4.8	4.51	4.6	2.4	7.05	1.166
EXTB	Pyrene	6	4.3	5.3	6.8	4.9	0.32	2.38	4.4	2.9	8.35	5	5.3	5	4.58	4.95	0.32	8.35	2.057
EXTB	Benzylbutylphthalate	6	4.2	1.8	0.5	0	5.12	1.66	4.8	4.1	2.88	5	3.1	5.5	3.22	3.6	0	5.5	1.872
EXTB	Bis(2-ethylhexylphthalate)	6	4.9	5	4.9	13.3	5.89	2.66	3.9	4.2	7.99	5	6.8	7.2	5.98	5	2.66	13.3	2.740
EXTB	Di-n-butylphthalate	6	4.5	3.9	2.7	1.9	6.12	2.22	4.4	0	7.49	6	5.2	7.1	4.29	4.45	0	7.49	2.264
EXTB	4-Bromophenyl phenyl ether	6	4.1	4.8	4.6	4.6	5.95	2.4	3.4	0.4	6.28	5	4.5	4.7	4.23	4.6	0.4	6.28	1.577
EXTB	4-chlorophenyl phenyl ether	6	4.4	5	4.9	3.8	5.43	2.54	3.8	3.2	6.49	7	5	4.4	4.66	4.65	2.54	7	1.277
EXTB	Bis(2-chloroethyl)ether	6	4.7	6.1	4.8	3.5	5.18	2.56	NA	2.3	5.91	6	4	4.3	4.49	4.7	2.3	6.1	1.315
EXTB	2,4-Dinitrotoluene	6	3.4	4.9	2.9	4.3	3.59	0.087	3.6	0	4.51	2	2	5.2	3.04	3.5	0	5.2	1.726
EXTB	2,6-Dinitrotoluene	6	4.1	4.8	3.2	5.6	3.41	1.32	3.2	3.7	4.57	4	3.1	2.3	3.61	3.55	1.32	5.6	1.140
EXTB	Bis(2-chloroethoxy)methane	6	5	8.9	4.7	5	5.66	2.19	NA	2.5	5.73	6	3.9	4.8	4.94	5	2.19	8.9	1.806
EXTB	N-Nitrosodi-n-propylamine	6	4.9	6.2	4.2	4.7	3.94	2.14	2.7	9.5	6.11	6	3.4	4.8	4.88	4.75	2.14	9.5	1.947

TABLE 1 - INTERLABORATORY STUDY 89-5
BASE/NEUTRAL EXTRACTABLE ORGANICS (MISA GROUP 19)
RESULTS EXPRESSED IN ppb

DISTRIBUTED: MAY 2, 1989		DESIGN	LABORATORY NUMBER														RANGE		
SAMPLE	PARAMETERS	(ppb)	2001	2002	2003	2004	2005	2006	2007	2008	2010	2011	2012	2013	MEAN	MEDIAN	MIN	MAX	STD DEV
EXTC	Acenaphthene	21	15	17	14	16.2	10.9	7.44	9.2	28.5	23.2	15	15.3	20	15.98	15.15	7.44	28.5	5.855
EXTC	Anthracene	21	15	15.1	13.8	16	0.2	6.63	9.2	8	26.9	15	14.7	16	13.04	14.85	0.2	26.9	6.543
EXTC	Benzo(a)anthracene	21	12	15.6	16.2	23.5	5.66	6.28	11	17	20.1	19	12.6	18	14.75	15.9	5.66	23.5	5.413
EXTC	Benzo(a)pyrene	21	13	12.6	13.9	24.8	< DL	7.03	11	0	19.1	16	13.6	18	13.55	13.6	0	24.8	6.458
EXTC	Benzo(b)fluoranthene	21	13	16 *	17.9	28	18.21	8.79	10	20.6 *	18.4	22	15	20	17.33	18.05	8.79	28	5.294
EXTC	Benzo(g,h,i)perylene	21	11	16.9	20.9	26.4	< DL	11.03	11	0	18.9	16	13.1	22	15.20	16	0	26.4	7.138
EXTC	Benzo(k)fluoranthene	21	12	16 *	19.8	25.8	13.9	7.96	11	20.6 *	19.2 **	16	13.8	20	16.34	16	7.96	25.8	4.967
EXTC	2-Chloronaphthalene	21	0	15.9	12.6	20.7	16.19	7.63	8.4	9.6	23.2	18	11.9	19	13.59	14.25	0	23.2	6.541
EXTC	Chrysene	21	12	18.5	19.2	25.9	18.61	6.82	11	15.2	16.4	16	12	18	15.80	16.2	6.82	25.9	4.926
EXTC	Dibenzo(a,h)anthracene	21	11	15.2	18.9	26.2	16.5	10.67	10	0	19.2	17	14.8	19	14.87	15.85	0	26.2	6.503
EXTC	Fluoranthene	21	15	17.1	29	21.5	20.7	10.32	11	12.1	34	15	17.7	30	19.45	17.4	10.32	34	7.845
EXTC	Fluorene	21	15	14.7	14.7	16.5	20.12	8.16	9.6	0	24.5	10	17.3	11	13.47	14.7	0	24.5	6.309
EXTC	Naphthalene	21	15	20.7	13.8	20.7	18.05	6.55	8	13.7	21.7	21	10.1	20	15.78	16.5	6.55	21.7	5.398
EXTC	Phenanthrene	21	15	17.7	16.6	18.5	20.09	9.96	11	10.3	28.9	14	18.4	22	16.87	17.15	9.96	28.9	5.429
EXTC	Pyrene	21	15	18	33.4	22	9.11	8.4	11	11	33.4	14	18.5	18	17.65	16.5	8.4	33.4	8.439
EXTC	Benzylbutylphthalate	21	16	6	1.4	25.6	23.94	8.08	12	26.6	10.2	15	12	21	14.82	13.5	1.4	26.6	8.081
EXTC	Bis(2-ethylhexylphthalate)	21	16	16.8	30.5	21.2	24.66	15.27	8.4	28.1	30.1	15	49.5	21	23.04	21.1	8.4	49.5	10.742
EXTC	Di-n-butylphthalate	21	15	13.8	11.3	17.3	22.87	8.88	10	1.1	22.8	16	17.3	21	14.78	15.5	1.1	22.87	6.329
EXTC	4-Bromophenyl phenyl ether	21	15	17	15.1	18.2	22.88	9.16	10	35.2	26.5	15	17.5	22	18.63	17.25	9.16	35.2	7.217
EXTC	4-chlorophenyl phenyl ether	21	15	17.4	14.9	16.8	20.37	8.89	8.4	7.1	25.2	20	18.1	21	16.10	17.1	7.1	25.2	5.571
EXTC	Bis(2-chloroethyl)ether	21	15	23	15.2	25.3	17.04	9.04	NA	11.1	21.4	17	9.6	21	16.79	17	9.04	25.3	5.477
EXTC	2,4-Dinitrotoluene	21	8.7	13.4	15.5	26.8	21.37	4.71	14	0	29.2	13	14.2	27	15.66	14.1	0	29.2	9.042
EXTC	2,6-Dinitrotoluene	21	11	16.7	15.3	25.9	27.2	6.89	13	11.8	28.3	16	13.5	11	16.38	13.2	6.89	28.3	7.005
EXTC	Bis(2-chloroethoxy)methane	21	16	20.9	15.9	25.9	21.68	7.84	NA	22.8	21.5	17	10.8	23	18.48	20.9	7.84	25.9	5.537
EXTC	N-Nitrosodi-n-propylamine	21	15	17.8	12.8	26.6	19.75	8.02	6	26.2	21	16	10.3	23	16.87	16.9	6	26.6	6.782

* Benzo(b)fluoranthene coelutes with benzo(k)flouranthene. The total result is divided equally between both parameters.
** Quantitated as Benzo(b)fluoranthene

IF THE RESULTS WERE REPORTED AS <MDL OR ND, 0 WAS ENTERED IF THE MDL IS LESS THAN THE SPIKE. OTHERWISE, <MDL (VALUE IF KNOWN) WAS ENTERED.

TABLE 2 - INTERLABORATORY STUDY 89-5
BASE/NEUTRAL EXTRACTABLE ORGANICS
RESULTS EXPRESSED AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MAY 2, 1989

SAMPLE	PARAMETERS	LABORATORY NUMBER														RANGE		
		DESIGN	2001	2002	2003	2004	2005	2006	2007	2008	2010	2011	2012	2013	MEAN	MEDIAN	MIN	MAX	STD DEV
EXTB	Acenaphthene	6	72%	83%	78%	62%	18%	37%	63%	167%	104%	83%	80%	72%	77%	75%	18%	167%	36%
EXTB	Anthracene	6	62%	67%	60%	60%		31%	63%	50%	110%	83%	83%	67%	67%	63%	31%	110%	20%
EXTB	Benzo(a)anthracene	6	57%	73%	72%	73%		24%	73%	35%	102%	100%	58%	83%	68%	73%	24%	102%	24%
EXTB	Benzo(a)pyrene	6	53%	55%	72%	77%		24%	73%	0%	71%	83%	45%	67%	56%	67%	0%	83%	25%
EXTB	Benzo(b)fluoranthene	6	62%	80%*	78%	80%	59%	31%	73%	187%*	87%	117%	48%	93%	69%	76%	18%	117%	27%
EXTB	Benzo(g,h,i)perylene	6	50%	85%	90%	90%		30%	67%	0%	48%	67%	38%	90%	59%	67%	0%	90%	29%
EXTB	Benzo(k)fluoranthene	6	47%	80%*	102%	87%	23%	26%	73%	187%*	84%**	83%	35%	88%	62%	77%	18%	102%	30%
EXTB	2-Chloronaphthalene	6	0%	82%	60%	73%	72%	35%	60%	187%	100%	100%	67%	68%	75%	70%	0%	187%	44%
EXTB	Chrysene	6	45%	95%	90%	78%	50%	26%	73%	73%	93%	83%	42%	85%	70%	76%	26%	95%	23%
EXTB	Dibenzo(a,h)anthracene	6	50%	72%	75%	88%	49%	24%	67%	0%	24%	67%	40%	88%	54%	59%	0%	88%	28%
EXTB	Fluoranthene	6	72%	83%	98%	83%	88%	46%	73%	50%	137%	83%	80%	20%	76%	82%	20%	137%	29%
EXTB	Fluorene	6	72%	95%	78%	90%	91%	38%	67%	0%	104%	67%	75%	38%	68%	74%	0%	104%	29%
EXTB	Naphthalene	6	75%	145%	70%	67%	85%	32%	55%	87%	95%	117%	70%	73%	81%	74%	32%	145%	29%
EXTB	Phenanthrene	6	68%	80%	73%	73%	84%	46%	73%	40%	118%	83%	82%	80%	75%	77%	40%	118%	19%
EXTB	Pyrene	6	72%	88%	113%	82%	5%	40%	73%	48%	139%	83%	88%	83%	76%	83%	5%	139%	34%
EXTB	Benzyl butyl phthalate	6	70%	30%	8%	0%	85%	28%	80%	68%	48%	83%	52%	92%	54%	60%	0%	92%	31%
EXTB	Bis(2-ethylhexyl)phthalate	6	82%	83%	82%	222%	98%	44%	65%	70%	133%	83%	113%	120%	100%	83%	44%	222%	46%
EXTB	Di-n-butylphthalate	6	75%	65%	45%	32%	102%	37%	73%	0%	125%	100%	87%	118%	72%	74%	0%	125%	38%
EXTB	4-Bromophenyl phenyl ether	6	68%	80%	77%	77%	99%	40%	57%	7%	105%	83%	75%	78%	70%	77%	7%	105%	26%
EXTB	4-chlorophenyl phenyl ether	6	73%	83%	82%	63%	91%	42%	63%	53%	108%	117%	83%	73%	78%	78%	42%	117%	21%
EXTB	Bis(2-chloroethyl)ether	6	78%	102%	80%	58%	86%	43%		38%	99%	100%	67%	72%	75%	78%	38%	102%	22%
EXTB	2,4-Dinitrotoluene	6	57%	82%	48%	72%	60%	1%	60%	0%	75%	33%	33%	87%	51%	59%	0%	87%	29%
EXTB	2,6-Dinitrotoluene	6	68%	80%	53%	93%	57%	22%	53%	62%	76%	67%	52%	38%	60%	60%	22%	93%	19%
EXTB	Bis(2-chloroethoxy)methane	6	83%	148%	78%	83%	94%	37%		42%	96%	100%	65%	80%	82%	83%	37%	148%	30%
EXTB	N-Nitrosodi-n-propylamine	6	82%	103%	70%	78%	66%	36%	45%	158%	102%	100%	57%	80%	81%	79%	36%	158%	32%
EXTC	Acenaphthene	21	71%	81%	67%	77%	52%	35%	44%	136%	110%	71%	73%	95%	76%	72%	35%	136%	28%
EXTC	Anthracene	21	71%	72%	66%	76%	1%	32%	44%	38%	128%	71%	70%	76%	62%	71%	1%	128%	31%
EXTC	Benzo(a)anthracene	21	57%	74%	77%	112%	27%	30%	52%	81%	96%	90%	60%	86%	70%	76%	27%	112%	26%
EXTC	Benzo(a)pyrene	21	62%	60%	66%	118%		33%	52%	0%	91%	76%	65%	86%	65%	65%	0%	118%	31%
EXTC	Benzo(b)fluoranthene	21	62%	76%*	85%	133%	87%	42%	48%	98%*	88%	105%	71%	95%	83%	86%	42%	133%	25%
EXTC	Benzo(g,h,i)perylene	21	52%	80%	100%	126%		53%	52%	0%	90%	76%	62%	105%	72%	76%	0%	126%	34%
EXTC	Benzo(k)fluoranthene	21	57%	76%*	94%	123%	66%	38%	52%	98%*	91%**	76%	66%	95%	78%	76%	38%	123%	24%
EXTC	2-Chloronaphthalene	21	0%	76%	60%	99%	77%	36%	40%	46%	110%	86%	57%	90%	65%	68%	0%	110%	31%
EXTC	Chrysene	21	57%	88%	91%	123%	89%	32%	52%	72%	78%	76%	57%	86%	75%	77%	32%	123%	23%
EXTC	Dibenzo(a,h)anthracene	21	52%	72%	90%	125%	79%	51%	48%	0%	91%	81%	70%	90%	71%	76%	0%	125%	31%
EXTC	Fluoranthene	21	71%	81%	138%	102%	99%	49%	52%	58%	162%	71%	84%	143%	93%	83%	49%	162%	37%
EXTC	Fluorene	21	71%	70%	70%	79%	96%	39%	46%	0%	117%	48%	82%	52%	64%	70%	0%	117%	30%
EXTC	Naphthalene	21	71%	99%	66%	99%	86%	31%	38%	65%	103%	100%	48%	95%	75%	79%	31%	103%	26%
EXTC	Phenanthrene	21	71%	84%	79%	88%	96%	47%	52%	49%	138%	67%	88%	105%	80%	82%	47%	138%	26%
EXTC	Pyrene	21	71%	86%	159%	105%	43%	40%	52%	52%	159%	67%	88%	86%	84%	79%	40%	159%	40%
EXTC	Benzyl butyl phthalate	21	76%	29%	7%	122%	114%	38%	57%	127%	49%	71%	57%	100%	71%	64%	7%	127%	38%
EXTC	Bis(2-ethylhexyl)phthalate	21	76%	80%	145%	101%	117%	73%	40%	134%	143%	71%	236%	100%	110%	101%	40%	236%	51%
EXTC	Di-n-butylphthalate	21	71%	66%	54%	82%	109%	42%	48%	5%	109%	76%	82%	100%	70%	74%	5%	109%	30%
EXTC	4-Bromophenyl phenyl ether	21	71%	81%	72%	87%	109%	44%	48%	168%	126%	71%	83%	105%	89%	82%	44%	168%	34%
EXTC	4-chlorophenyl phenyl ether	21	71%	83%	71%	80%	97%	42%	40%	34%	120%	95%	86%	100%	77%	82%	34%	120%	27%
EXTC	Bis(2-chloroethyl)ether	21	71%	110%	72%	120%	81%	43%		53%	102%	81%	46%	100%	80%	81%	43%	120%	26%
EXTC	2,4-Dinitrotoluene	21	41%	64%	74%	128%	102%	22%	67%	0%	139%	62%	68%	129%	75%	68%	0%	139%	43%
EXTC	2,6-Dinitrotoluene	21	52%	80%	73%	123%	130%	33%	62%	56%	135%	76%	64%	52%	78%	69%	33%	135%	33%
EXTC	Bis(2-chloroethoxy)methane	21	76%	100%	76%	123%	103%	37%		109%	102%	81%	51%	110%	88%	100%	37%	123%	26%
EXTC	N-Nitrosodi-n-propylamine	21	71%	85%	61%	127%	94%	38%	29%	125%	100%	76%	49%	110%	80%	81%	29%	127%	32%

* Benzo(b)fluoranthene coelutes with benzo(k)flouranthene; The total result is divided equally between two parameters

** Quantitated as Benzo(b)fluoranthene

TABLE 3 - INTERLABORATORY STUDY 89-5
ACID EXTRACTABLE ORGANICS (MISA GROUP 20)
RESULTS EXPRESSED IN ppb

DISTRIBUTED: MAY 2, 1989		DESIGN	LABORATORY NUMBER													RANGE			
SAMPLE	PARAMETERS	(ppb)	2001	2002	2003	2004	2005	2006	2007	2008	2010	2011	2012	2013	MEAN	MEDIAN	MIN	MAX	STD DEV
EXTA	2,3,4,5-Tetrachlorophenol																		
EXTA	2,3,4,6-Tetrachlorophenol																		
EXTA	2,3,5,6-Tetrachlorophenol																		
EXTA	2,3,5-Trichlorophenol																		
EXTA	2,4,5-Trichlorophenol																		
EXTA	2,4,6-Trichlorophenol																		
EXTA	2,4-Dimethylphenol																		
EXTA	2,4-Dichlorophenol																		
EXTA	4,6-Dinitro-o-cresol																		
EXTA	2-Chlorophenol																		
EXTA	4-Chloro-3-methylphenol																		
EXTA	4-Nitrophenol																		
EXTA	p-Cresol																		
EXTA	Pentachlorophenol																		
EXTA	Phenol																		
EXTB	2,3,4,5-Tetrachlorophenol	6	5.1	4.6 **	2.6	6.3 ††	5.65	3.72	2.2 ††	6.2	4.65	9	4.9	6.4	5.11	5	2.20	9.00	1.832
EXTB	2,3,4,6-Tetrachlorophenol	6	4.0	4.6 **	3.1	6.3 ††	3.96	3.05	2.2 ††	14.5	3.07	3	4.5	4.6	4.74	3.98	2.20	14.50	3.258
EXTB	2,3,5,6-Tetrachlorophenol	6	2.8	4.6 **	2.6	4.5	3.21	5.2	2.6	1.9	0.52	2	3.4	5.6	3.24	3.01	0.52	5.60	1.496
EXTB	2,3,5-Trichlorophenol	6		ND	ND	0.9	< DL	3.65	2.6	6.8	4.45 @@	5 *		6.9	4.33	3.65	0.90	6.90	2.682
EXTB	2,4,5-Trichlorophenol	6	6.2	4.6	3.4	5	2.7	2.41	3.2	5.3	2.79	3	4.4	<MDL	3.91	3.4	2.41	6.20	1.251
EXTB	2,4,6-Trichlorophenol	6	5.2	5.3	3.7	1.5	2.07	1.65	3.4	5.7	3.27	5	5.8	5.4	4.00	4.35	1.50	5.80	1.615
EXTB	2,4-Dimethylphenol	6	< 7.3	3	1	2.2	0.63	0	NA	26.7	6.34	8	3.7	1.6	5.32	2.6	0.00	26.70	7.546
EXTB	2,4-Dichlorophenol	6	6.2	7.9	3.8	5.7	1.84	1.65	1.8	5.1	4.36	3	4.8	4.2	4.20	4.28	1.65	7.90	1.920
EXTB	4,6-Dinitro-o-cresol	6	<24.0	4.6	1.5	< 16	1.37	< 9.3	2.6	&&	0	3	0	150 @	1.87	2.6	0.00	4.60	1.666
EXTB	2-Chlorophenol	6	6.8	4.7	4	5.7	3.07	0	NA	5.1	4.7	7	5	9.1	5.02	5	0.00	9.10	2.334
EXTB	4-Chloro-3-methylphenol	6	6.4	4.2	4.1	0.2	0.6	1.54	4.6	34.7	4.15	5	4.4	3.9	6.15	4.18	0.20	34.70	9.177
EXTB	4-Nitrophenol	6	5.2	2.5	1.7	0	0.9	0	1.7	7.2	0	0.75	0	4.7	2.05	1.3	0.00	7.20	2.432
EXTB	p-Cresol	6	7.8	3.6	2.7	0.6		1.93	4	1.3	0.37	6	3.9	3.7	3.26	3.6	0.37	7.80	2.252
EXTB	Pentachlorophenol	6	4.3	4.5	3.5	3.1	2.89	1.39	2.4	6	0	1	6.1	11	3.85	3.3	0.00	11.00	2.925
EXTB	Phenol	6	6.8	2.1	2.3	0	3.02	0	NA	4.2	2.35	4	3.5	3.1	2.58	3.02	0.00	6.80	2.006
EXTB	2,3,4-Trichlorophenol		5.7	4.5			2.69						6	4.9	4.76	4.9	2.69	6.00	1.303
EXTC	2,3,4,5-Tetrachlorophenol	21	18.0	11.6 **	12.7	12.3 ††	37.11	15.43	6.5 ††	18.8	34.8	46	18.1	17	20.70	17.5	6.50	46.00	12.009
EXTC	2,3,4,6-Tetrachlorophenol	21	17.0	11.6 **	10.9	12.3 ††	28.73	13.42	6.5 ††	41.4	25.4	9	19.1	12	17.28	12.86	6.50	41.40	10.034
EXTC	2,3,5,6-Tetrachlorophenol	21	12.0	11.6 **	10.8	26.1	26.68	24.96	7.2	15.9	19.4	8	15.4	15	16.09	15.2	7.20	26.68	6.829
EXTC	2,3,5-Trichlorophenol	21		ND	ND	29.4	< DL	13.61	7.2	26.3	31.9 @@	10 *	< 1	17	19.34	17	7.2	31.9	11.388
EXTC	2,4,5-Trichlorophenol	21	20.0	16.1	15.7	28.5	15.81	9.48	9	50.5	34.1	13	18.4	<MDL	20.96	16.1	9	50.5	12.352
EXTC	2,4,6-Trichlorophenol	21	23.0	17.2	15.1	31.3	14.27	6.45	9.2	17.6	33.8	15	21.9	12	18.07	16.15	6.45	33.8	8.233
EXTC	2,4-Dimethylphenol	21	26.0	8.9	7.9	16.2	0.87	2.57	NA	78.1	37	30	15.4	2.5	18.79	15.4	0.87	78.1	22.537
EXTC	2,4-Dichlorophenol	21	25.0	20.4	14.3	29.4	10.74	6.02	6.4	14.7	33.2	10	19.5	7.5	16.43	14.5	6.02	33.2	9.136
EXTC	4,6-Dinitro-o-cresol	21	<24.0	18.2	15.7	80.2	14.94	0	7.4	&&	36.6	18	10	370 @	22.34	16.85	0	370	23.876
EXTC	2-Chlorophenol	21	28.0	17.8	14.1	26.1	12.65	5.05	NA	15.3	30.3	21	20.2	3.7	16.18	17.8	3.7	30.3	8.676
EXTC	4-Chloro-3-methylphenol	21	23.0	16.1	15.8	29.3	3.97	5.97	14	101.7	32	17	19.6	7.1	23.80	16.55	3.97	101.7	26.007
EXTC	4-Nitrophenol	21	18.0	7.3	7.5	6.2	17.7	0.8	8.2	38.8	42	5	5.1	13	14.13	7.85	0.8	42	13.299
EXTC	p-Cresol	21	25.0	10.2	11	11.3		8.17	15	12.1	23.6	20	17.2	4.3	14.35	12.1	4.3	25	6.492
EXTC	Pentachlorophenol	21	19.0	17.5	21.6	36.3	13.6	9.05	8	22.2	47.6	6	28.2	30	21.59	20.3	6	47.6	12.358
EXTC	Phenol	21	29.0	8.9	7.8	24.5	12.51	1.54	NA	12.5	15.4	12	14.1	0.6	11.57	12.5	0.6	29	8.509
EXTC	2,3,4-Trichlorophenol		20.0	17			14.91						28.8	12	18.54	17	12.00	28.80	6.437

* Results based on 2,3,4-trichlorophenol.
** The three tetrachlorophenol co-elute, therefore the total result is divided equally between the 3 parameters.
†† 2,3,4,5- & 2,3,4,6-Tetrachlorophenol coelute, therefore the total result reported is divided equally between the two parameters.
@@ 2,3,5-trichlorophenol different retention time than standard quantitated.
&& MDL under review.
@ RESULT EXCLUDED FROM CALCULATION OF MEAN, MEDIAN AND STANDARD DEVIATION
IF THE RESULTS WERE REPORTED AS <MDL OR ND, 0 WAS ENTERED IF THE MDL IS LESS THAN THE SPIKE. OTHERWISE <MDL (VALUE IF KNOWN) WAS ENTERED.
VALUES REPORTED AS "< VALUE" EXCLUDED FROM STATISTICAL CALCULATIONS.

TABLE 4 - INTERLABORATORY STUDY 89-5
ACID EXTRACTABLE ORGANICS (MISA GROUP 20)
RESULTS EXPRESSED AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MAY 2, 1989		DESIGN	LABORATORY NUMBER														RANGE		STD
SAMPLE	PARAMETERS	(ppb)	2001	2002	2003	2004	2005	2006	2007	2008	2010	2011	2012	2013	MEAN	MEDIAN	MIN	MAX	DEV
EXTB	2,3,4,5-Tetrachlorophenol	6	85%	77%**	43%	105%++	94%	62%	37%++	103%	78%	150%	82%	107%	85%	83%	37%	150%	31%
EXTB	2,3,4,6-Tetrachlorophenol	6	67%	77%**	52%	105%++	66%	51%	37%++	242%	51%	50%	75%	77%	79%	66%	37%	242%	54%
EXTB	2,3,5,6-Tetrachlorophenol	6	47%	77%**	43%	75%	54%	87%	43%	32%	9%	33%	57%	93%	54%	50%	9%	93%	25%
EXTB	2,3,5-Trichlorophenol	6				15%		61%	43%	113%	74%@@	33%*		115%	65%	61%	15%	115%	39%
EXTB	2,4,5-Trichlorophenol	6	103%	77%	57%	83%	45%	40%	53%	88%	47%	50%	73%		65%	57%	40%	103%	21%
EXTB	2,4,6-Trichlorophenol	6	87%	88%	62%	25%	35%	28%	57%	95%	55%	83%	97%	90%	67%	73%	25%	97%	27%
EXTB	2,4-Dimethylphenol	6		50%	17%	37%	11%	0%	NA	445%	106%	133%	62%	27%	89%	43%	0%	445%	132%
EXTB	2,4-Dichlorophenol	6	103%	132%	63%	95%	31%	28%	30%	85%	73%	50%	80%	70%	70%	71%	28%	132%	32%
EXTB	4,6-Dinitro-o-cresol	6		77%	25%		23%		43%		0%	50%	0%	2500%@	31%	43%	0%	2500%	28%
EXTB	2-Chlorophenol	6	113%	78%	67%	95%	51%	0%	NA	85%	78%	117%	83%	152%	84%	83%	0%	152%	39%
EXTB	4-Chloro-3-methylphenol	6	107%	70%	68%	3%	10%	26%	77%	578%	69%	83%	73%	65%	102%	70%	3%	578%	153%
EXTB	4-Nitrophenol	6	87%	42%	28%	0%	15%	0%	28%	120%	0%	13%	0%	78%	34%	22%	0%	120%	40%
EXTB	p-Cresol	6	130%	60%	45%	10%		32%	67%	22%	6%	100%	65%	62%	54%	60%	0%	130%	38%
EXTB	Pentachlorophenol	6	72%	75%	58%	52%	48%	23%	40%	100%	0%	17%	102%	183%	64%	55%	0%	183%	49%
EXTB	Phenol	6	113%	35%	38%	0%	50%	0%	NA	70%	39%	67%	58%	52%	48%	50%	0%	113%	32%
EXTC	2,3,4,5-Tetrachlorophenol	21	86%	55%**	60%	59%++	177%	73%	31%++	90%	166%	219%	86%	81%	99%	83%	31%	219%	57%
EXTC	2,3,4,6-Tetrachlorophenol	21	81%	55%**	52%	59%++	137%	64%	31%++	197%	121%	43%	91%	57%	82%	61%	31%	197%	48%
EXTC	2,3,5,6-Tetrachlorophenol	21	57%	55%**	51%	124%	127%	119%	34%	76%	92%	38%	73%	71%	77%	72%	34%	127%	33%
EXTC	2,3,5-Trichlorophenol	21				140%		65%	34%	125%	152%@@	48%*		81%	92%	81%	34%	152%	47%
EXTC	2,4,5-Trichlorophenol	21	95%	77%	75%	136%	75%	45%	43%	240%	162%	62%	88%		100%	77%	43%	240%	59%
EXTC	2,4,6-Trichlorophenol	21	110%	82%	72%	149%	68%	31%	44%	84%	161%	71%	104%	57%	86%	79%	31%	161%	39%
EXTC	2,4-Dimethylphenol	21	124%	42%	38%	77%	4%	12%	NA	372%	176%	143%	73%	12%	98%	73%	4%	372%	107%
EXTC	2,4-Dichlorophenol	21	119%	97%	68%	140%	51%	29%	30%	70%	158%	48%	93%	36%	78%	69%	29%	158%	44%
EXTC	4,6-Dinitro-o-cresol	21		87%	75%	382%	71%	0%	35%		174%	86%	48%	1762%@	106%	80%	0%	1762%	114%
EXTC	2-Chlorophenol	21	133%	85%	67%	124%	60%	24%	NA	73%	144%	100%	96%	18%	84%	85%	-18%	144%	41%
EXTC	4-Chloro-3-methylphenol	21	110%	77%	75%	140%	19%	28%	67%	484%	152%	81%	93%	34%	113%	79%	19%	484%	124%
EXTC	4-Nitrophenol	21	86%	35%	36%	30%	84%	4%	39%	185%	200%	24%	24%	62%	67%	37%	4%	200%	63%
EXTC	p-Cresol	21	119%	49%	52%	54%		39%	71%	58%	112%	95%	82%	20%	68%	58%	0%	119%	31%
EXTC	Pentachlorophenol	21	90%	83%	103%	173%	65%	43%	38%	106%	227%	29%	134%	143%	103%	99%	29%	227%	59%
EXTC	Phenol	21	138%	42%	37%	117%	60%	7%	NA	60%	73%	57%	67%	3%	60%	59%	3%	138%	41%

* Results based on 2,3,4-Trichlorophenol.
** The three tetrachlorophenols co-elute, therefore the total result is divided equally between the 3 parameters.
++ 2,3,4,5- & 2,3,4,6-Tetrachlorophenol coelute, therefore the total result reported is divided equally between the two parameters.
@@ 2,3,5-trichlorophenol different retention time than standard quantitated.
@ RESULT EXCLUDED FROM CALCULATION OF MEAN AND MEDIAN

IF THE RESULTS WERE REPORTED AS <MDL OR ND, 0 WAS ENTERED IF THE MDL IS LESS THAN THE SPIKE. OTHERWISE <MDL (VALUE IF KNOWN) WAS ENTERED.

TABLE 5 - INTERLABORATORY STUDY 89-5
NEUTRAL CHLORINATED EXTRACTABLE ORGANICS
RESULTS EXPRESSED IN ppb

DISTRIBUTED: MAY 2, 1989		DESIGN	LABORATORY NUMBER													RANGE				
SAMPLE	PARAMETER	(ppb)	2001	2002	2003	2004	2005	2006	2007	2008	2010	2012	2013	MEAN	MEDIAN	MIN	MAX	STD DEV	2011	2003*
EXTA	1,2,3,4-Tetrachlorobenzene																			
EXTA	1,2,4,5-Tetrachlorobenzene																			
EXTA	1,2,3-Trichlorobenzene																			
EXTA	1,2,4-Trichlorobenzene																			
EXTA	Hexachlorobenzene		0.07																	
EXTA	Hexachlorobutadiene		0.05																	
EXTA	Hexachloroethane		0.1																	
EXTB	1,2,3,4-Tetrachlorobenzene	6	3.8	6	2.4	1	4.9	3.32	4.4	NA	4.25	4.3	3	3.74	4.03	1.00	6.00	1.394	1.5	
EXTB	1,2,4,5-Tetrachlorobenzene	6	1.7	6.3	3	1	5.1	4.72	4.5	NA	4	4.6	2.3	3.72	4.25	1.00	6.30	1.668	1.3	
EXTB	1,2,3-Trichlorobenzene	6	2.7	5.6	4.6	2.4	4.8	1.85	3.8	NA	4.46	4.3	3.9	3.84	4.1	1.85	5.60	1.180	1.36	
EXTB	1,2,4-Trichlorobenzene	6	<.01	7.1	2.6	2.5	4.59	0.96	4.6	3.2	4.82	3.8	5.4	3.96	4.2	0.96	7.1	1.725	1.5	
EXTB	Hexachlorobenzene	6	2.7	4.4	4.4	3.9	6.19	1.9	4.5	NA	6.69	4.3	2.9	4.19	4.35	1.90	6.69	1.478	0.54	4.6
EXTB	Hexachlorobutadiene	6	0.7	5.9	2.4	2.9 TR	4.28	1.07	3.8	NA	3.97	4.1	2	2.83	3.35	0.00	5.90	1.795	0.495	3.2
EXTB	Hexachloroethane	6	1.2	5.2	1.3	3.4 TR	3.89	0.87	3.6	2.5	4.25	2.9	6.9	3.16	3.4	0	6.9	1.983	0.37	3.2
EXTC	1,2,3,4-Tetrachlorobenzene	21	18.0	21	8.8	4.1	18.9	11.56	9.9	NA	17.7	12.8	14	13.68	13.4	4.10	21.00	5.280	1.64	
EXTC	1,2,4,5-Tetrachlorobenzene	21	10.0	25	11.5	4.7	19.7	16.78	11	NA	16.7	14.3	10	13.97	12.9	4.70	25.00	5.796	1.585	
EXTC	1,2,3-Trichlorobenzene	21	16.0	23	18.6	6.6	18.9	6.46	9.6	NA	16.7	9.9	23	14.88	16.35	6.46	23.00	6.312	1.725	
EXTC	1,2,4-Trichlorobenzene	21	<.01	17.8	10.8	7.1	16.45	3.19	11	21.3	18.8	8.7	30	14.51	13.7	3.19	30	7.862	2.03	
EXTC	Hexachlorobenzene	21	19.0	17.4	14.9	16.6	24.8	5.32	11	NA	24.2	14.8	15	16.30	15.8	5.32	24.80	5.752	0.615	15.9
EXTC	Hexachlorobutadiene	21	7.0	15.2	9.7	20.7	15.4	3.61	9.1	NA	13.9	7.3	7.5	10.94	9.4	3.61	20.70	5.178	0.47	10.3
EXTC	Hexachloroethane	21	11.0	14.7	5.6	16	12.53	3.55	9.8	9.8	13.6	4.8	41	13.14	11	3.55	41	10.634	0.49	10.4

TR Results qualified with a remark code.
* Additional set of results analyzed using GC/MS; Not included in statistical calculations.

NOTE: AFTER REVIEW OF THE DATA SET, DISTRIBUTED TO THE PARTICIPANTS SEPTEMBER 15, 1989, LABORATORY 2011 REQUESTED THAT THEIR DATA FOR MISA GROUP 23 BE WITHDRAWN. THEIR RESULTS ARE PROVIDED FOR INFORMATION PURPOSES ONLY AND ARE NOT INCLUDED IN THE STATISTICAL CALCULATIONS.

TABLE 6 - INTERLABORATORY STUDY 89-5
NEUTRAL CHLORINATED EXTRACTABLE ORGANICS (MISA GROUP 23)
RESULTS EXPRESSED AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MAY 2, 1989		DESIGN														RANGE		STD
SAMPLE	PARAMETERS	(ppb)	2001	2002	2003	2004	2005	2006	2007	2008	2010	2012	2013	MEAN	MEDIAN	MIN	MAX	DEV
EXTB	1,2,3,4-Tetrachlorobenzene	6	63%	100%	40%	17%	82%	55%	73%	NA	71%	72%	50%	62%	72%	17%	100%	23%
EXTB	1,2,4,5-Tetrachlorobenzene	6	28%	105%	50%	17%	85%	79%	75%	NA	67%	77%	38%	62%	71%	17%	105%	28%
EXTB	1,2,3-Trichlorobenzene	6	45%	93%	77%	40%	80%	31%	63%	NA	74%	72%	65%	64%	68%	31%	93%	20%
EXTB	1,2,4-Trichlorobenzene	6	0%	118%	43%	42%	77%	16%	77%	53%	80%	63%	90%	66%	70%	16%	118%	34%
EXTB	Hexachlorobenzene	6	45%	73%	73%	65%	103%	32%	75%	NA	112%	72%	48%	70%	73%	32%	112%	25%
EXTB	Hexachlorobutadiene	6	12%	98%	40%	48% TR	71%	18%	63%	NA	66%	68%	33%	47%	56%	0%	98%	30%
EXTB	Hexachloroethane	6	20%	87%	22%	57% TR	65%	15%	60%	42%	71%	48%	115%	50%	57%	0%	115%	33%
EXTC	1,2,3,4-Tetrachlorobenzene	21	86%	100%	42%	20%	90%	55%	47%	NA	84%	61%	67%	65%	64%	20%	100%	25%
EXTC	1,2,4,5-Tetrachlorobenzene	21	48%	119%	55%	22%	94%	80%	52%	NA	80%	68%	48%	67%	61%	22%	119%	28%
EXTC	1,2,3-Trichlorobenzene	21	76%	110%	89%	31%	90%	31%	46%	NA	80%	47%	110%	71%	78%	31%	110%	30%
EXTC	1,2,4-Trichlorobenzene	21	0%	85%	51%	34%	78%	15%	52%	101%	90%	41%	143%	69%	65%	15%	143%	41%
EXTC	Hexachlorobenzene	21	90%	83%	71%	79%	118%	25%	52%	NA	115%	70%	71%	78%	75%	25%	118%	27%
EXTC	Hexachlorobutadiene	21	33%	72%	46%	99%	73%	17%	43%	NA	66%	35%	36%	52%	45%	17%	99%	25%
EXTC	Hexachloroethane	21	52%	70%	27%	76%	60%	17%	47%	47%	65%	23%	195%	62%	52%	17%	195%	48%

FIG. 1: INTERLABORATORY STUDY 89-5

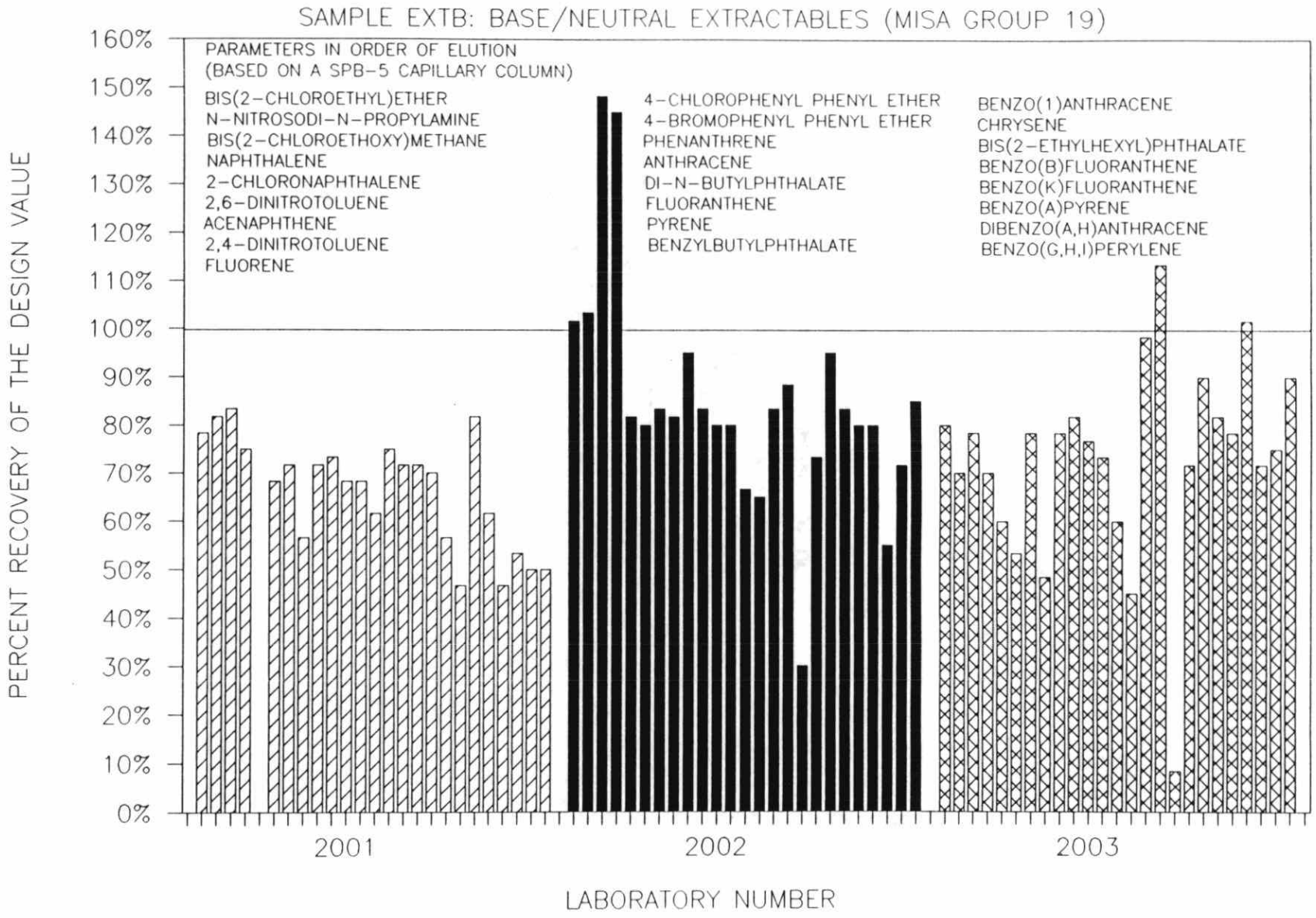


FIG. 2: INTERLABORATORY STUDY 89-5

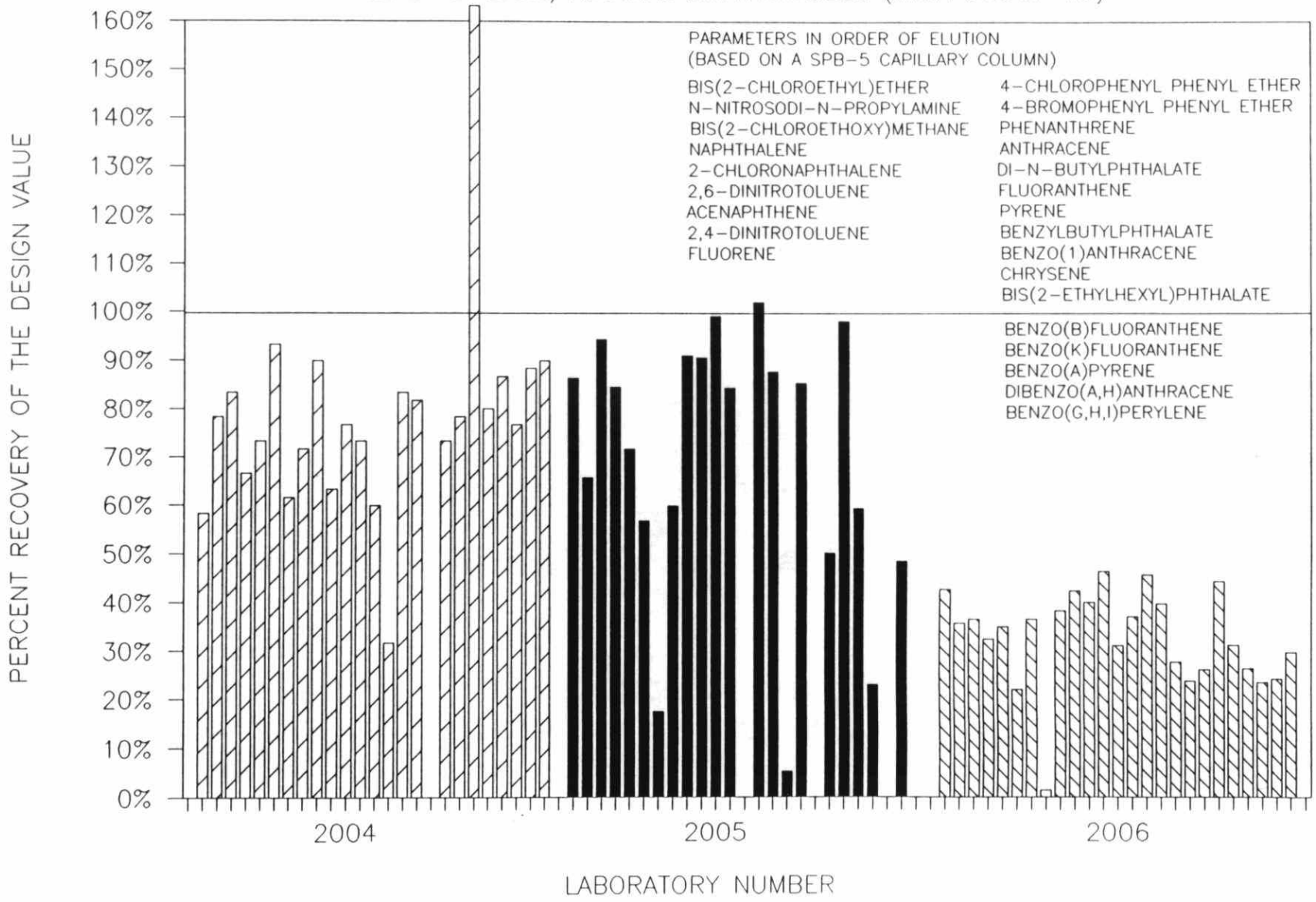


FIG. 3: INTERLABORATORY STUDY 89-5

SAMPLE EXTB: BASE NEUTRAL EXTRACTABLES (MISA GROUP 19)

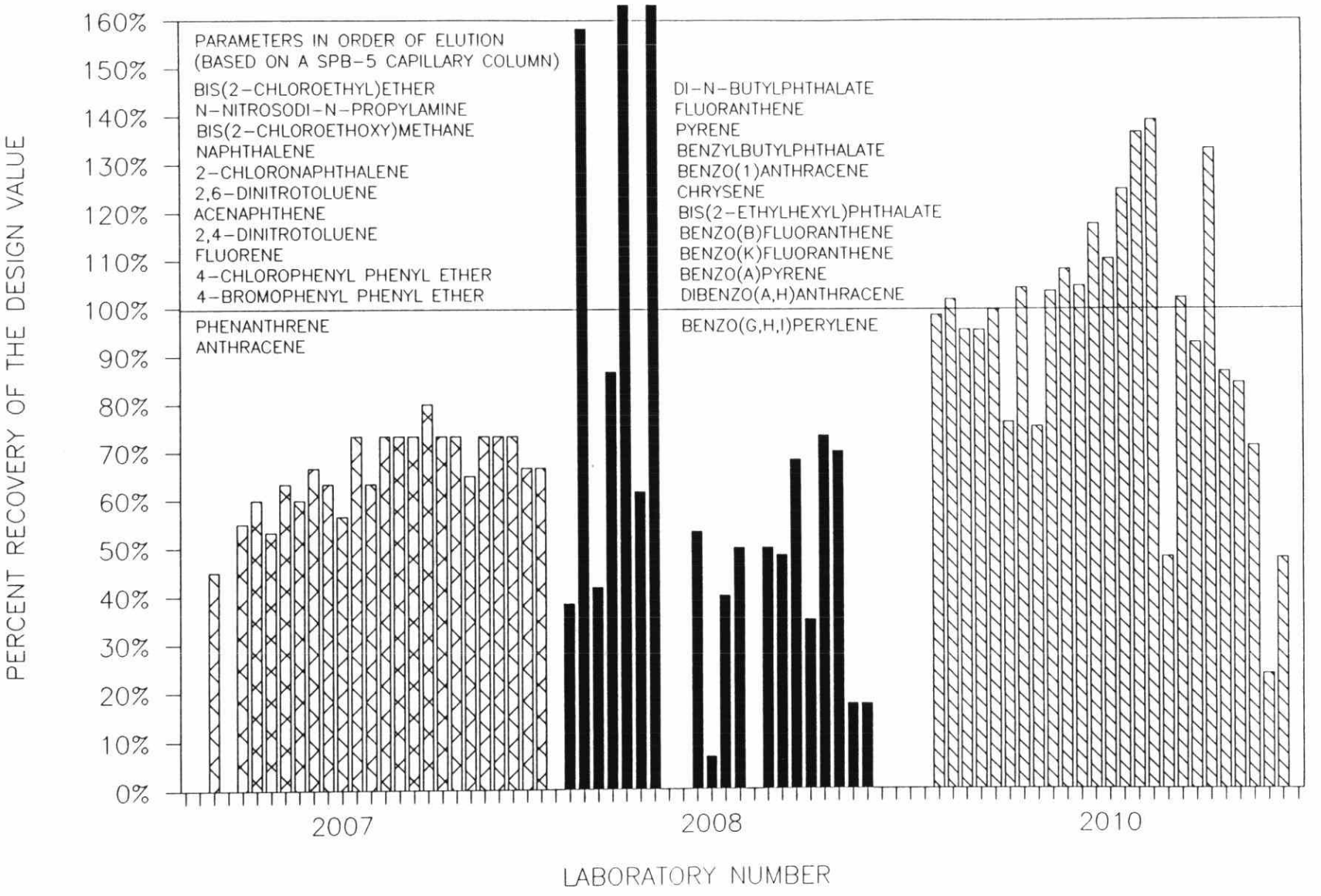


FIG. 4: INTERLABORATORY STUDY 89-5

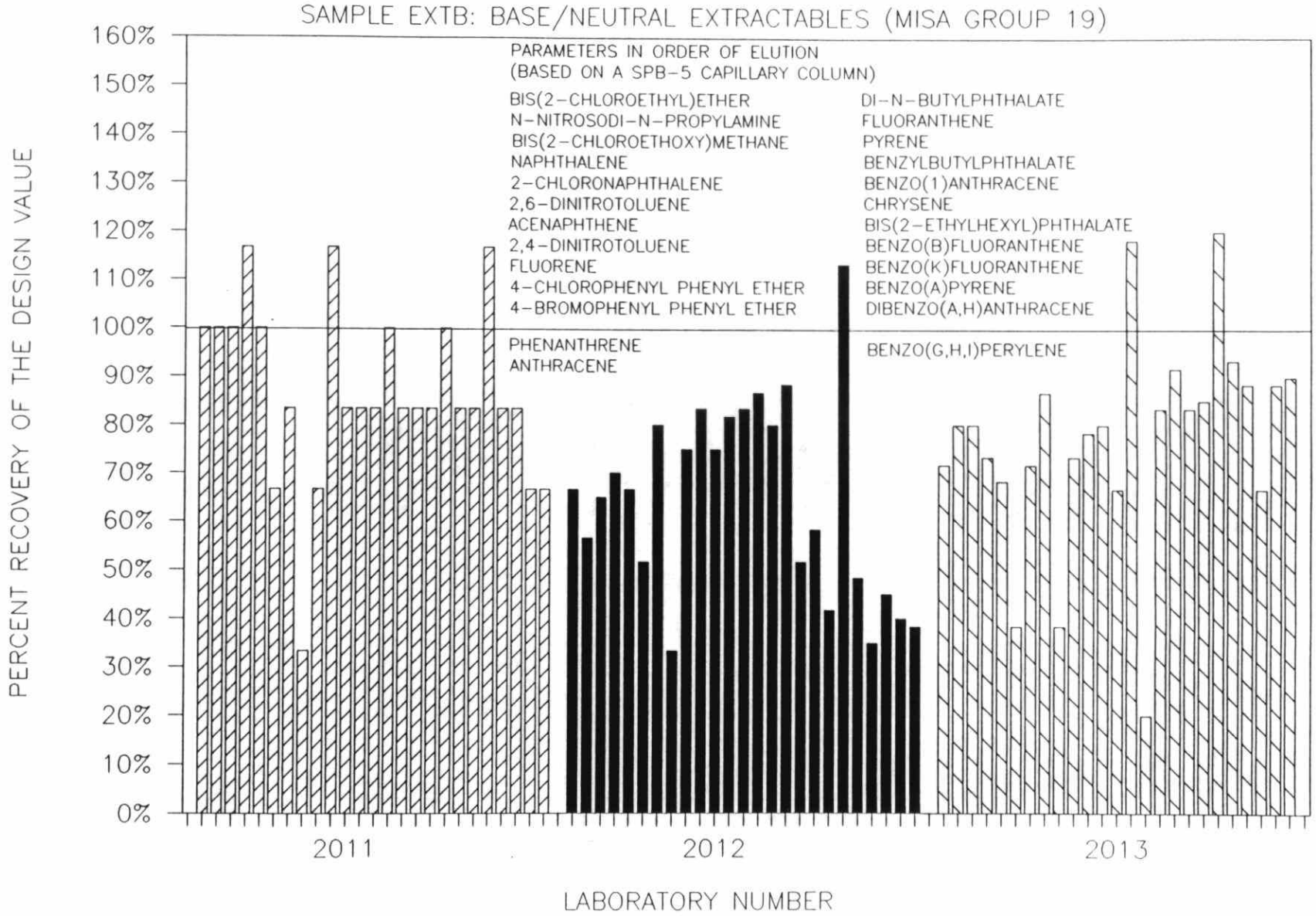


FIG. 5: INTERLABORATORY STUDY 89-5

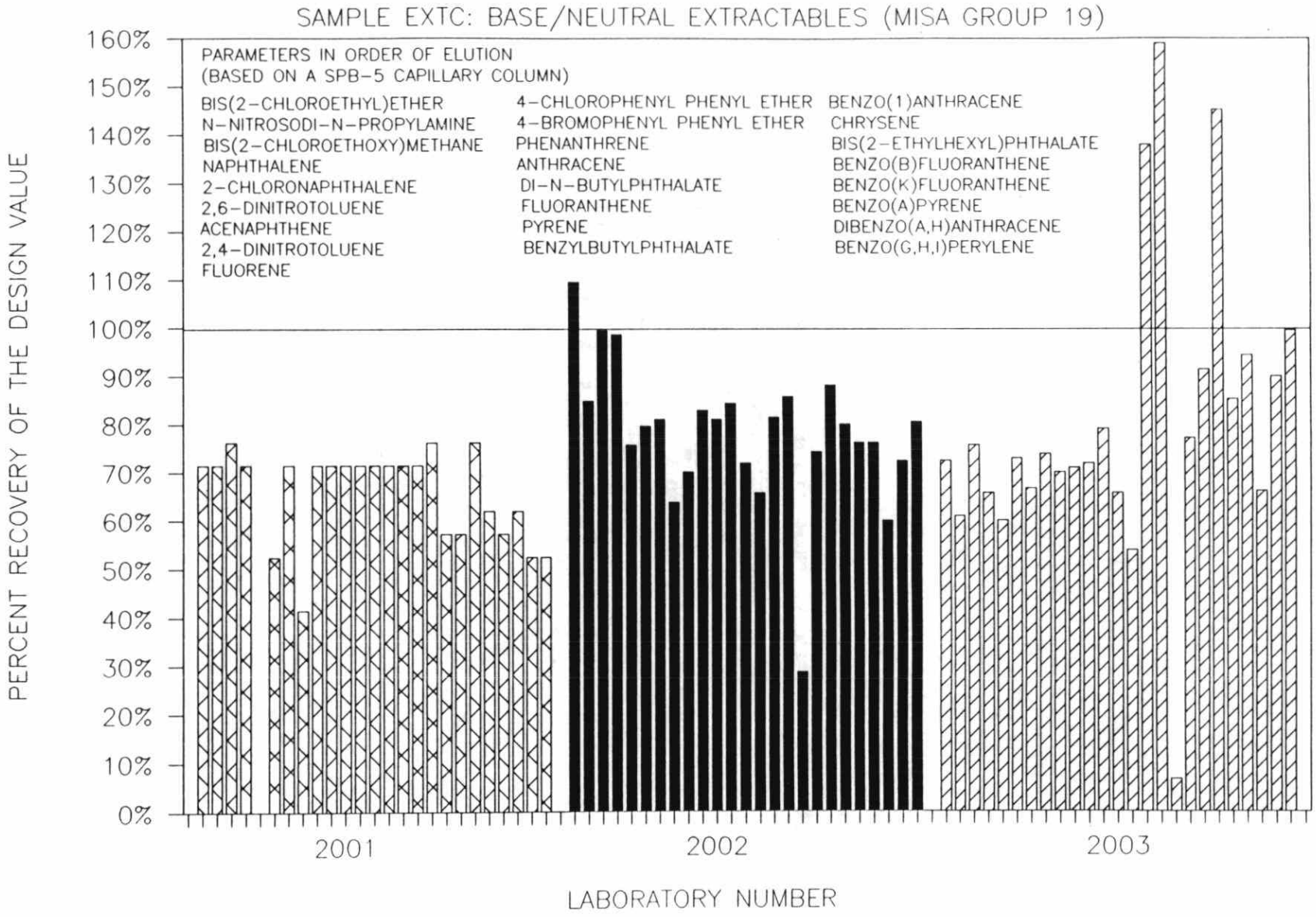


FIG. 6: INTERLABORATORY STUDY 89-5

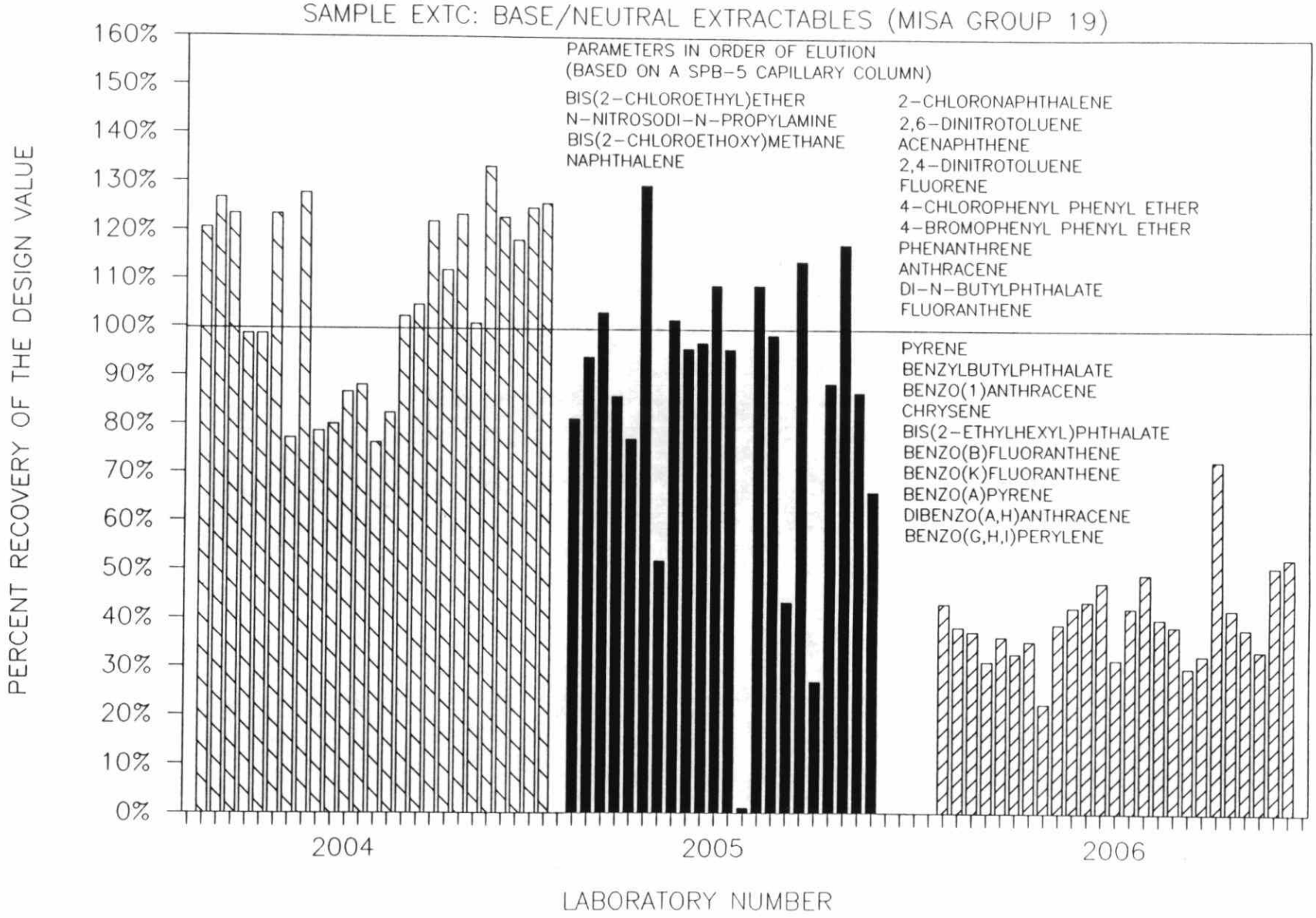


FIG. 7: INTERLABORATORY STUDY 89-5

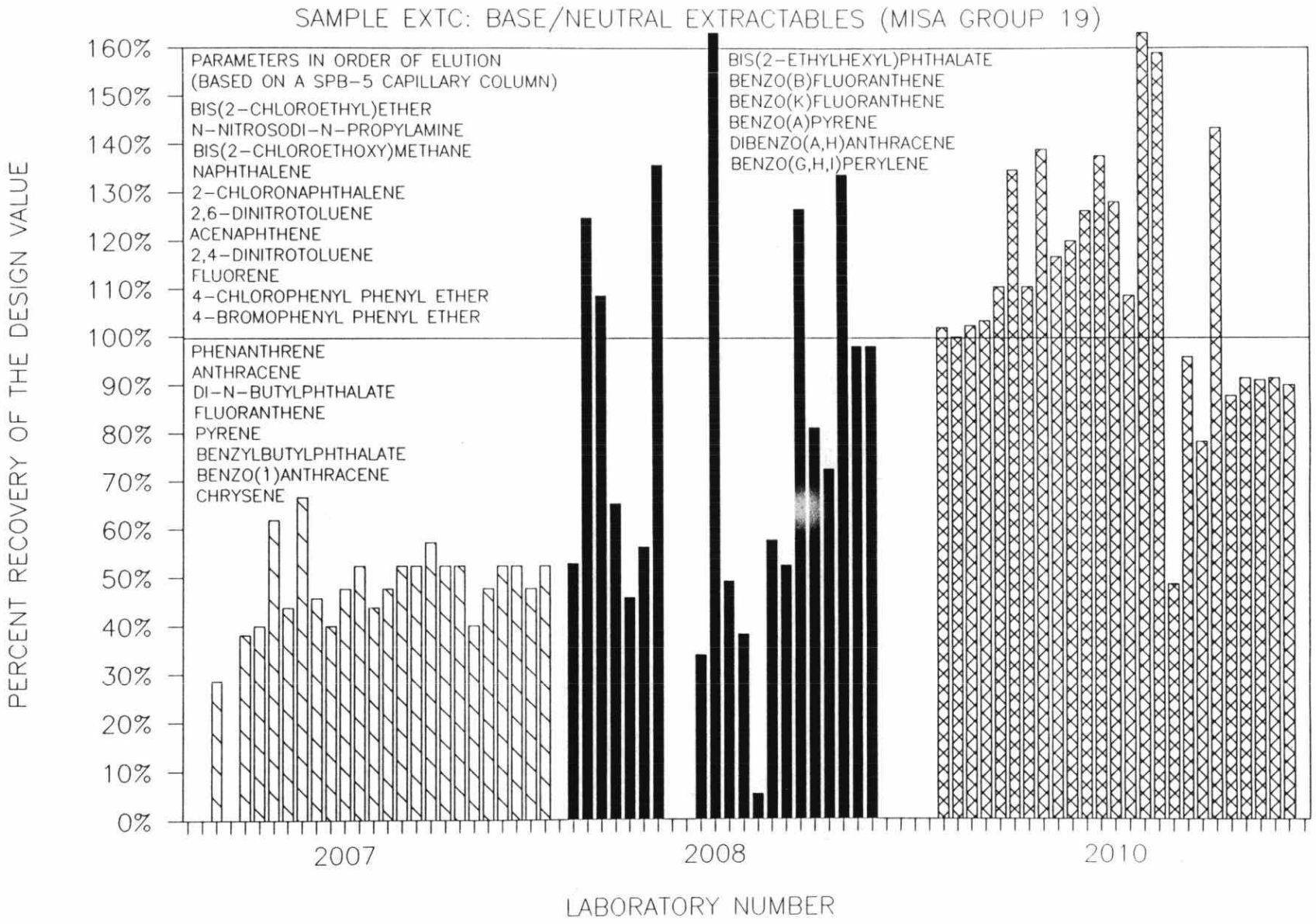


FIG. 8: INTERLABORATORY STUDY 89-5

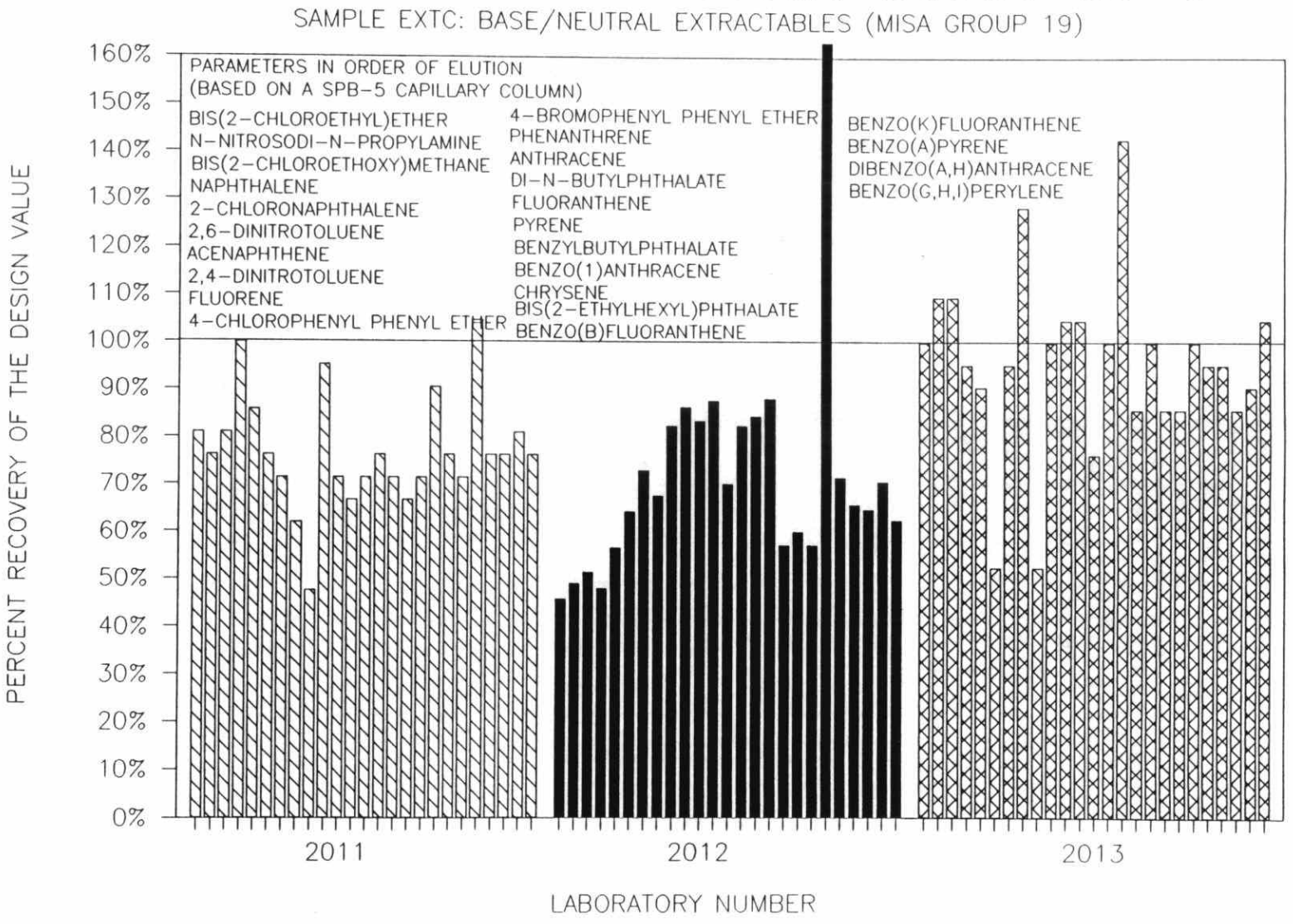


FIG. 9: INTERLABORATORY STUDY 89-5

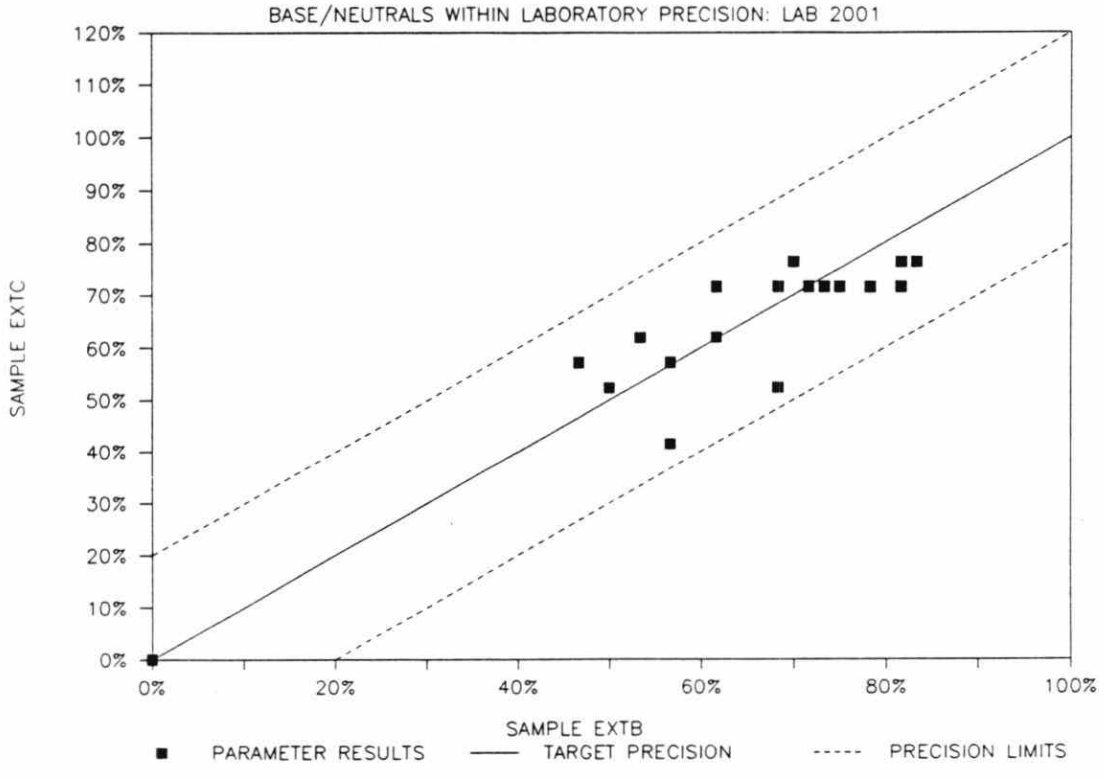


FIG. 10: INTERLABORATORY STUDY 89-5

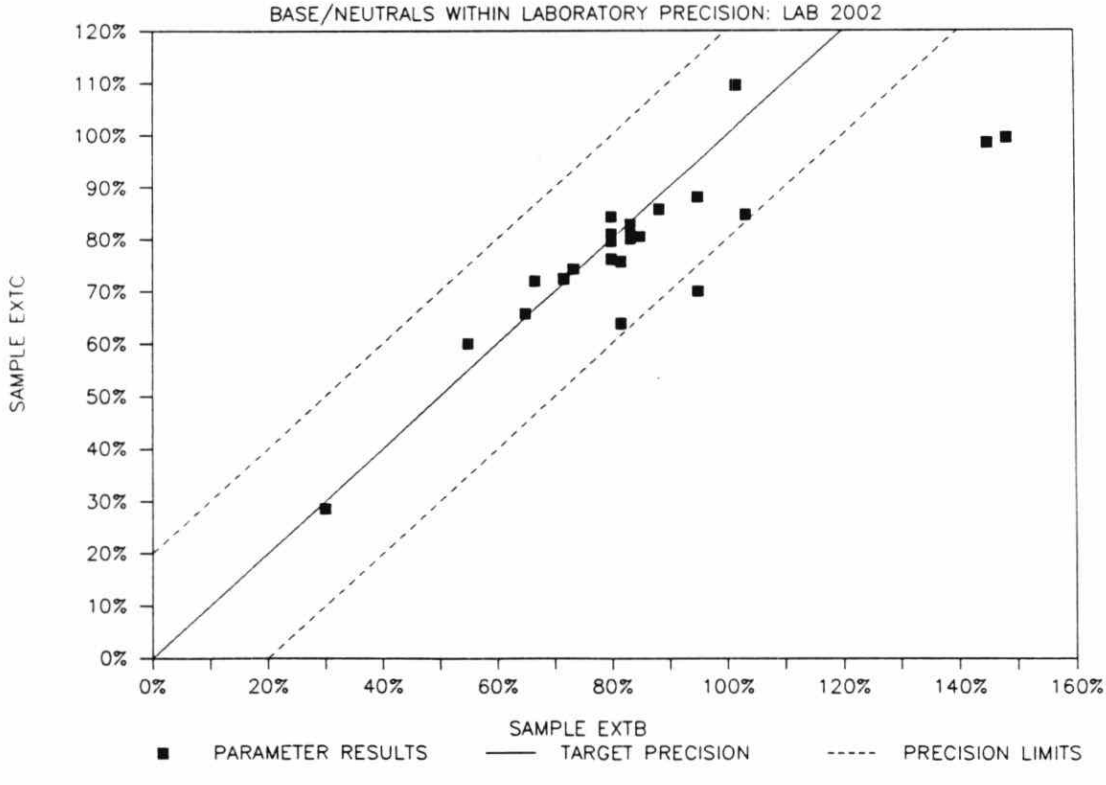


FIG. 11: INTERLABORATORY STUDY 89-5

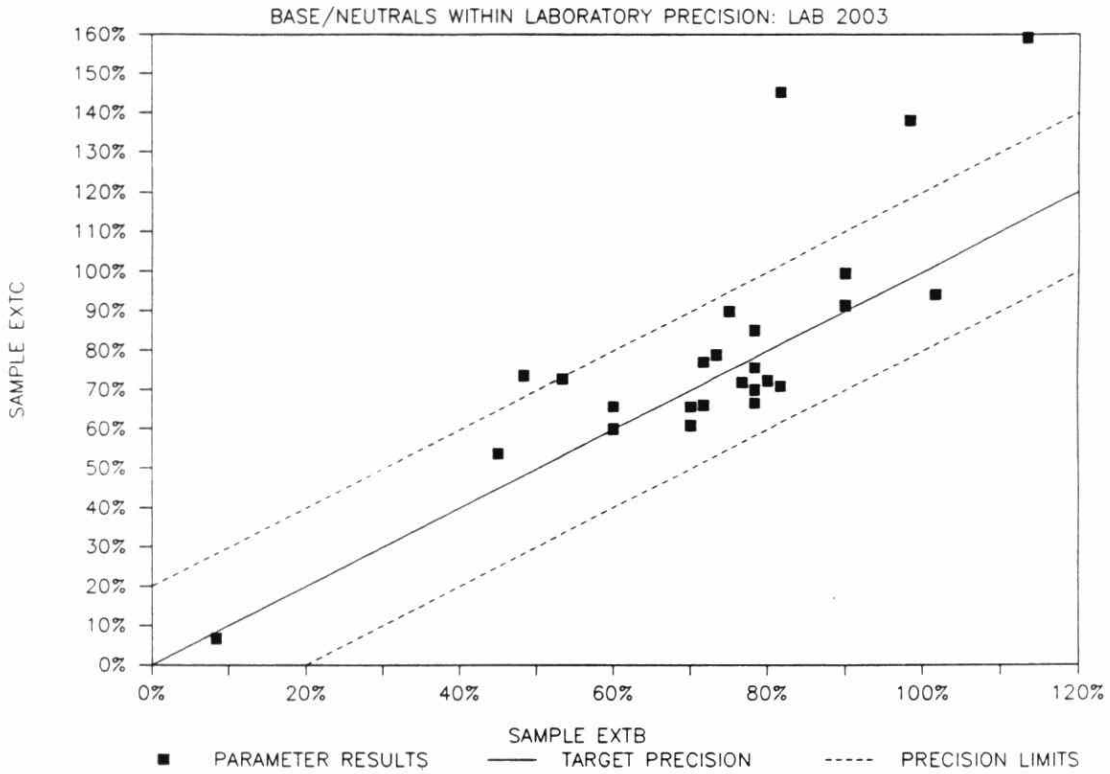


FIG. 12: INTERLABORATORY STUDY 89-5

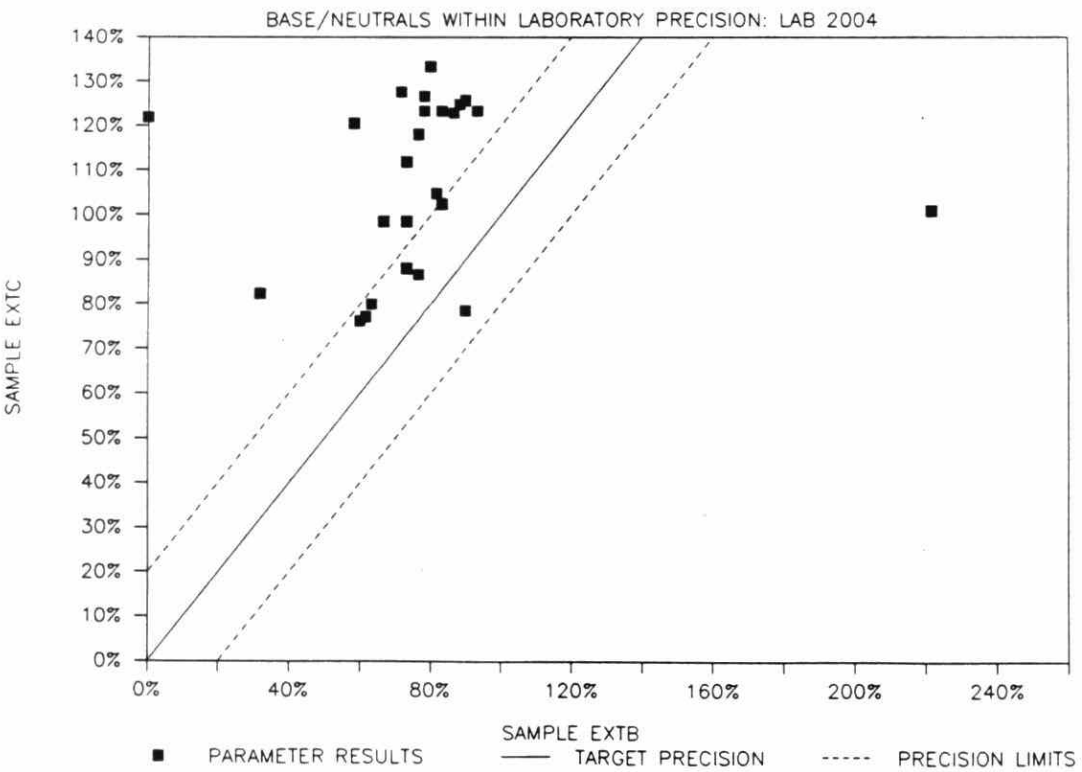


FIG. 13: INTERLABORATORY STUDY 89-5

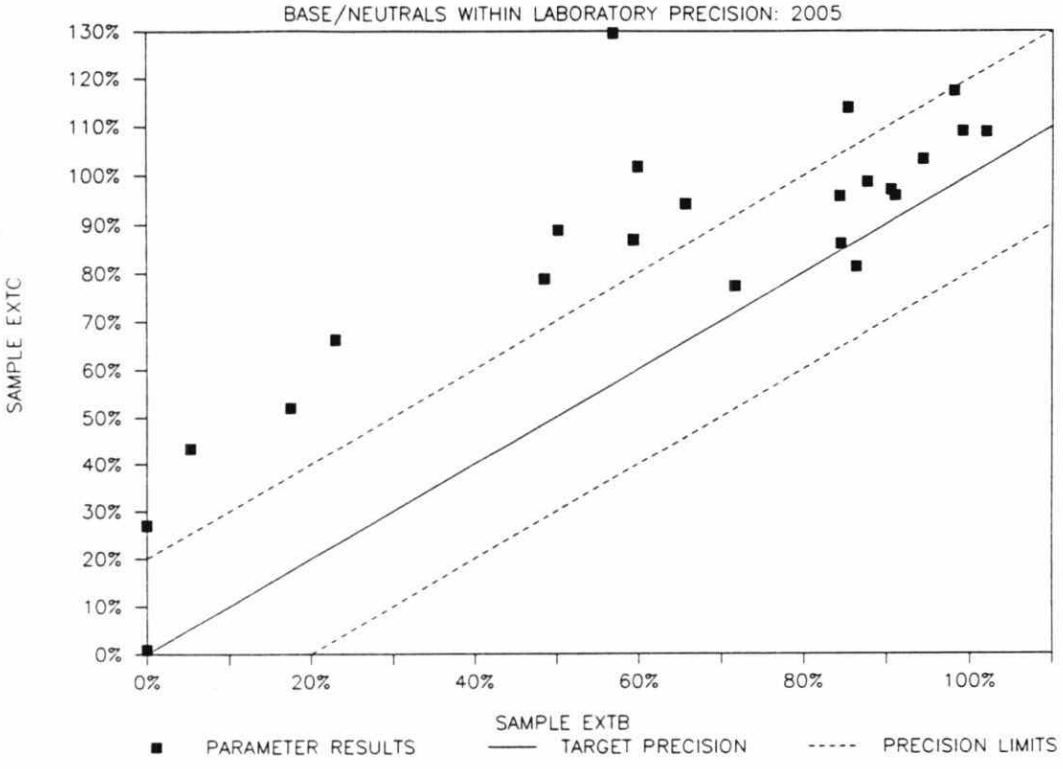


FIG. 14: INTERLABORATORY STUDY 89-5

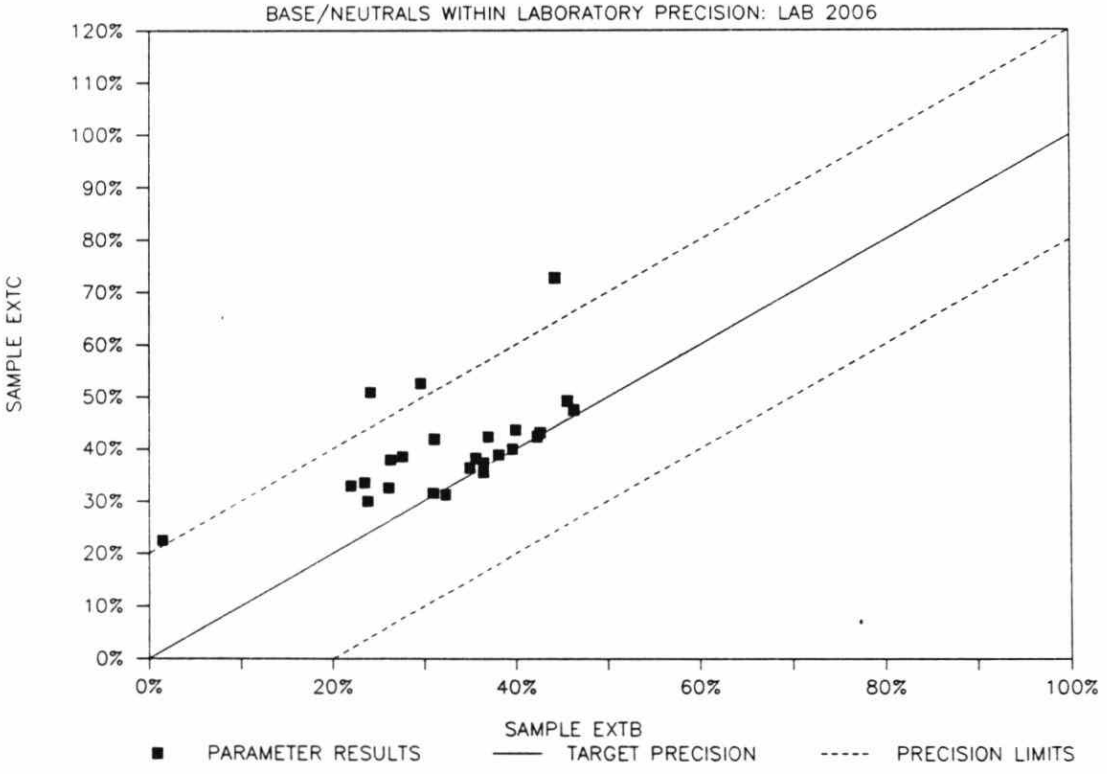


FIG. 15: INTERLABORATORY STUDY 89-5

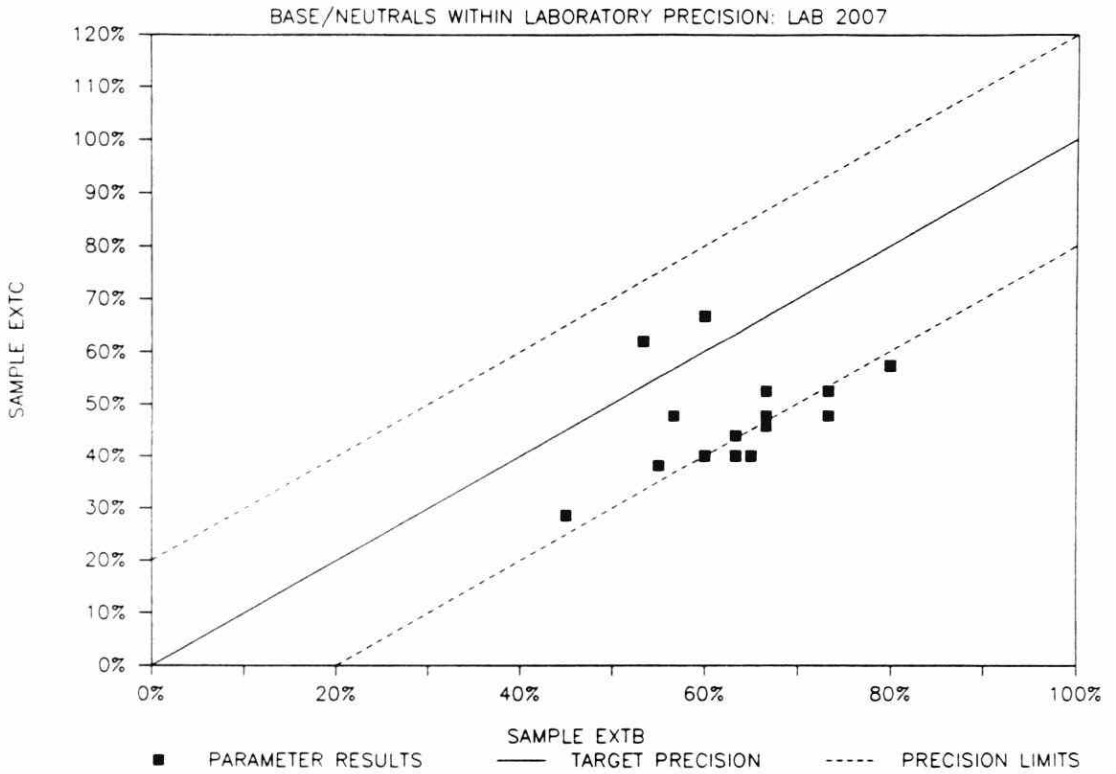


FIG. 16: INTERLABORATORY STUDY 89-5

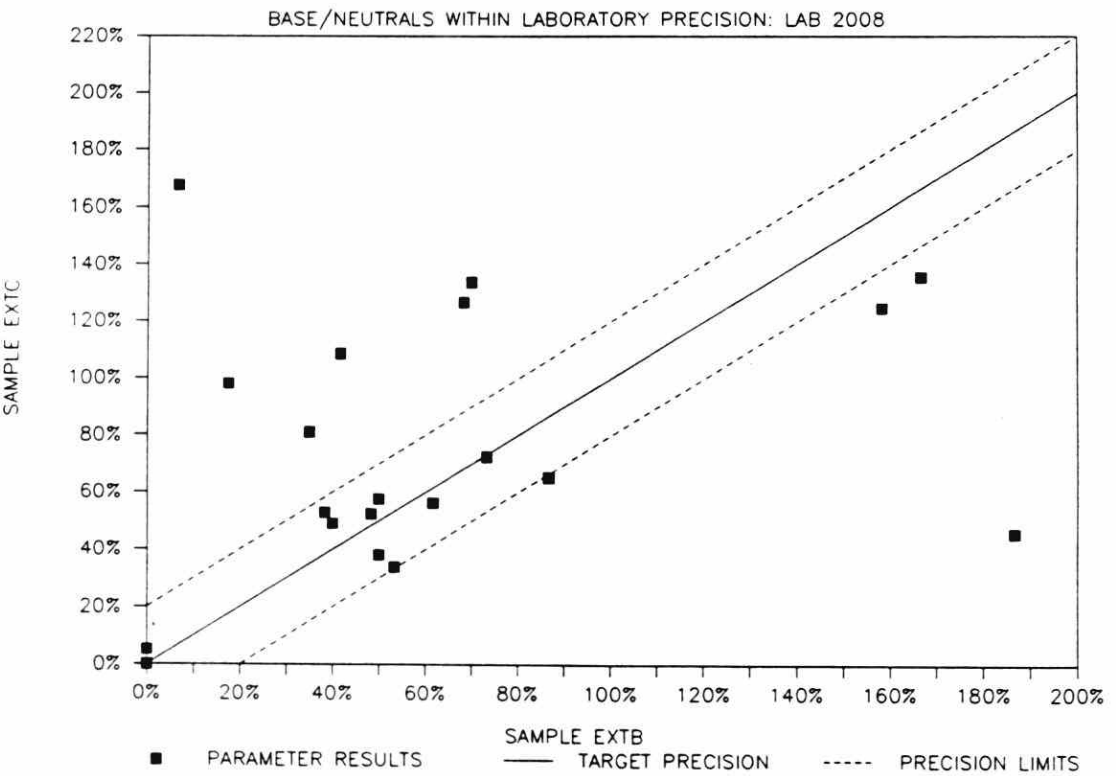


FIG. 17: INTERLABORATORY STUDY 89-5

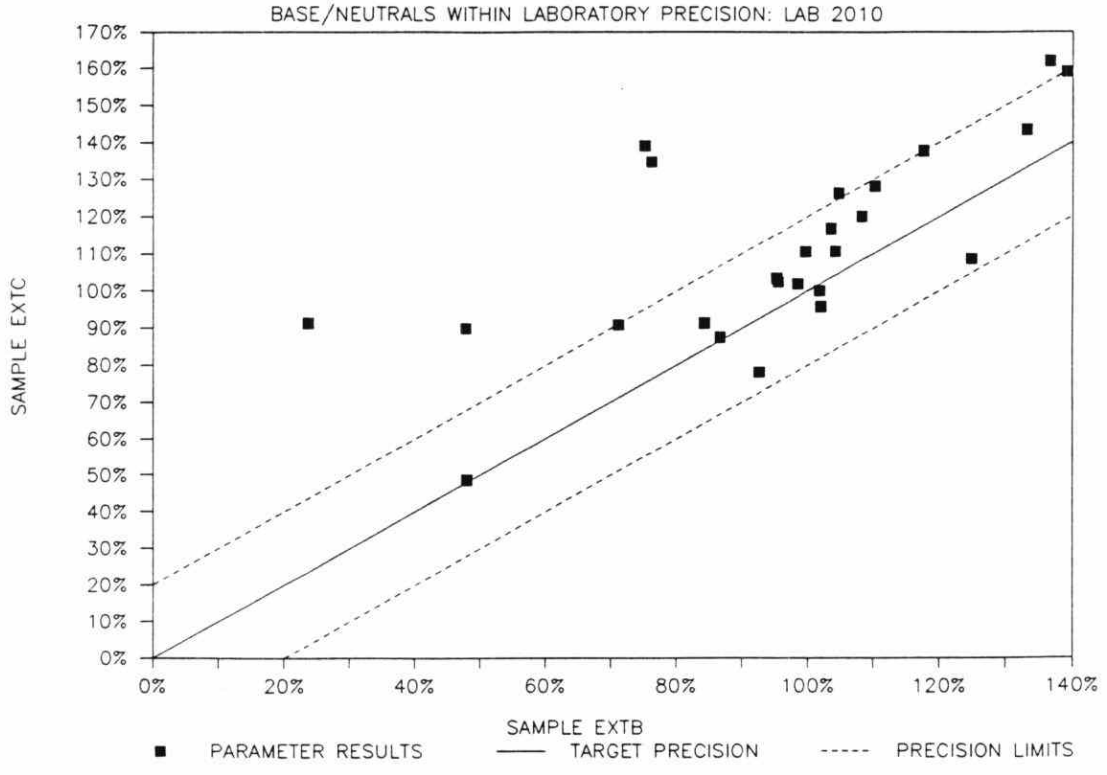


FIG. 18: INTERLABORATORY STUDY 89-5

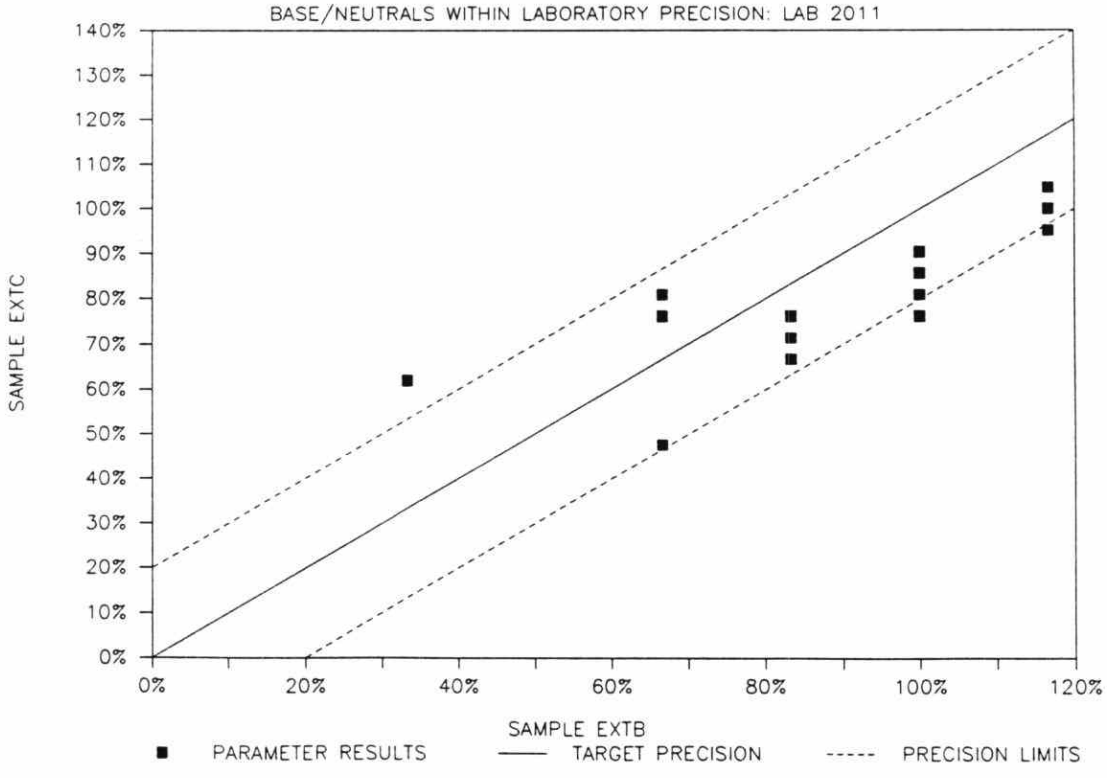


FIG. 19: INTERLABORATORY STUDY 89-5

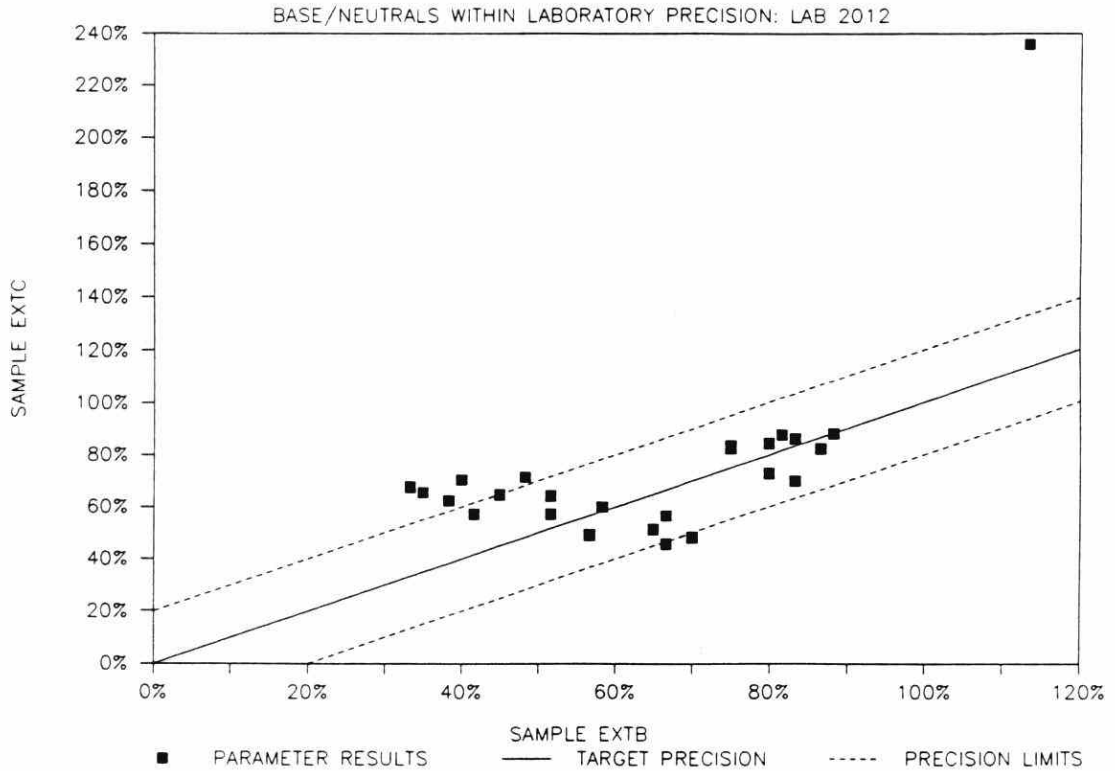


FIG. 20: INTERLABORATORY STUDY 89-5

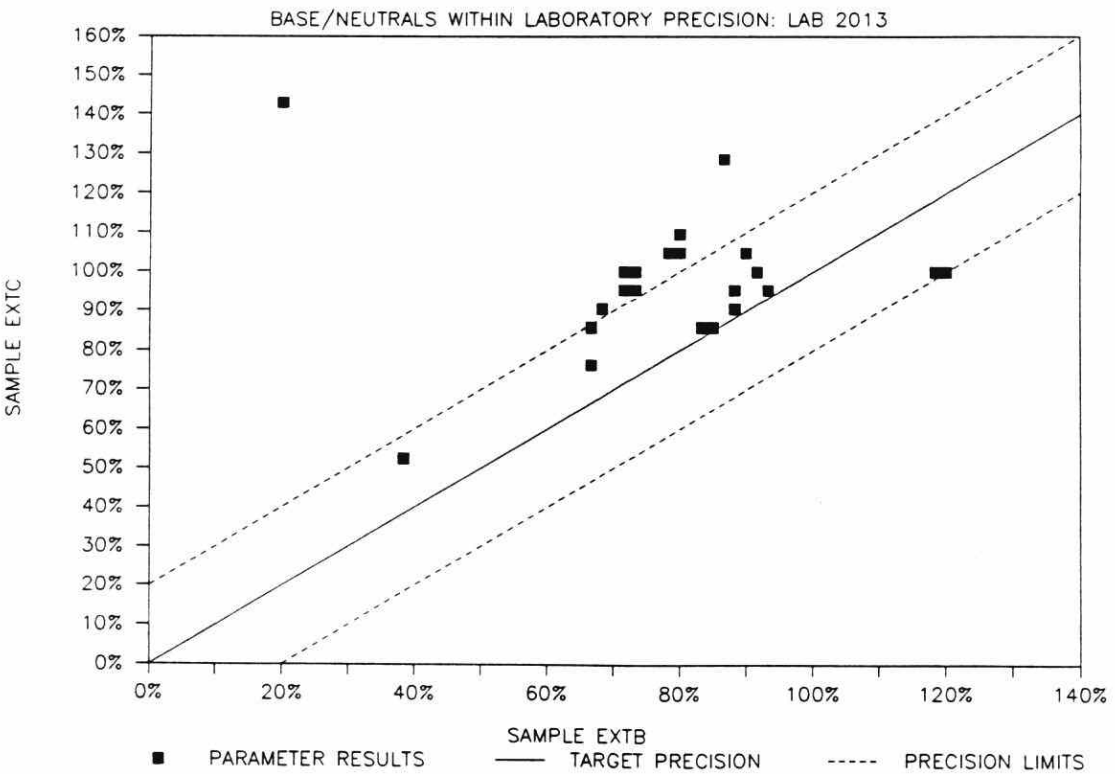


FIG. 21: INTERLABORATORY STUDY 89-5

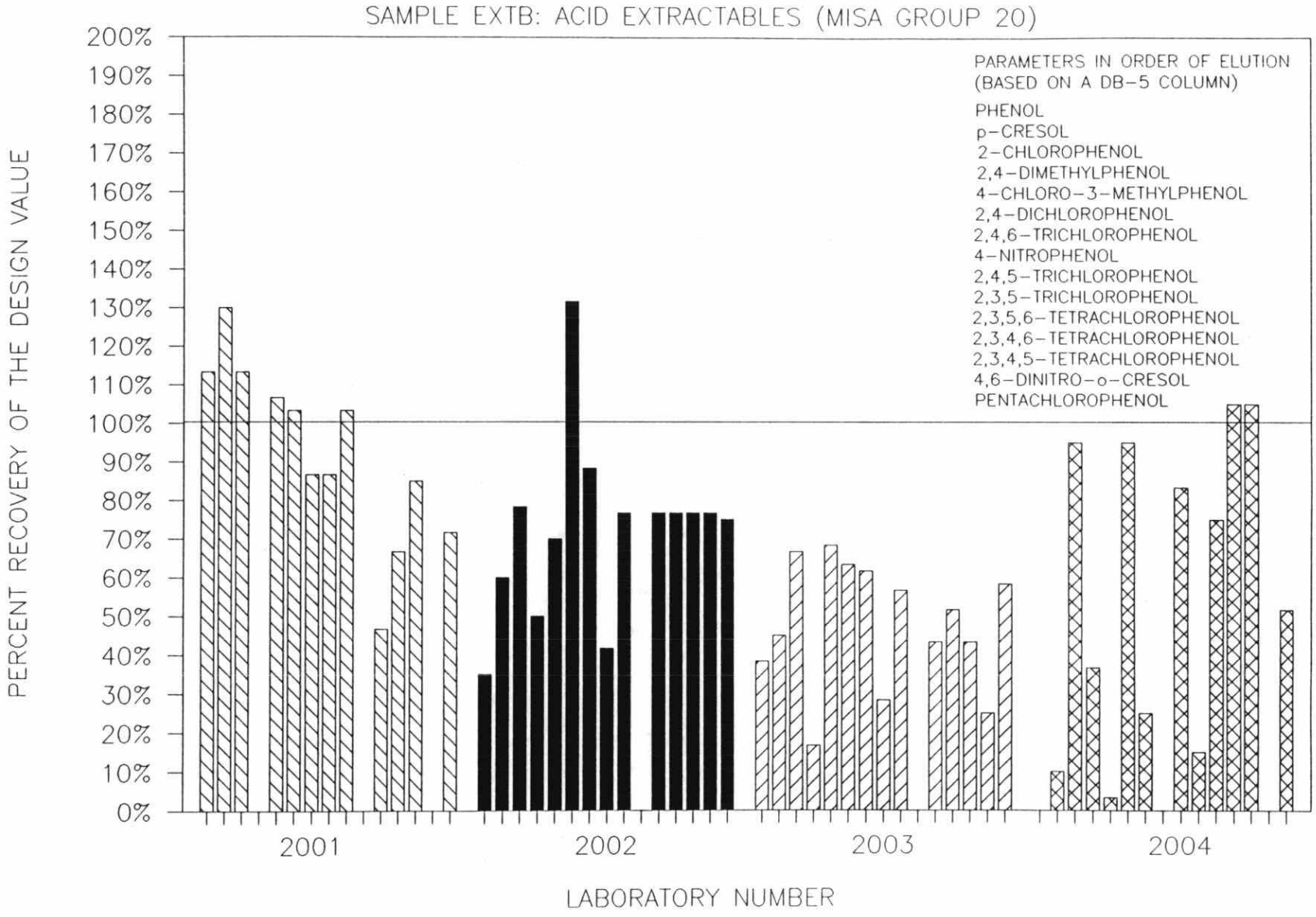


FIG. 22: INTERLABORATORY STUDY 89-5

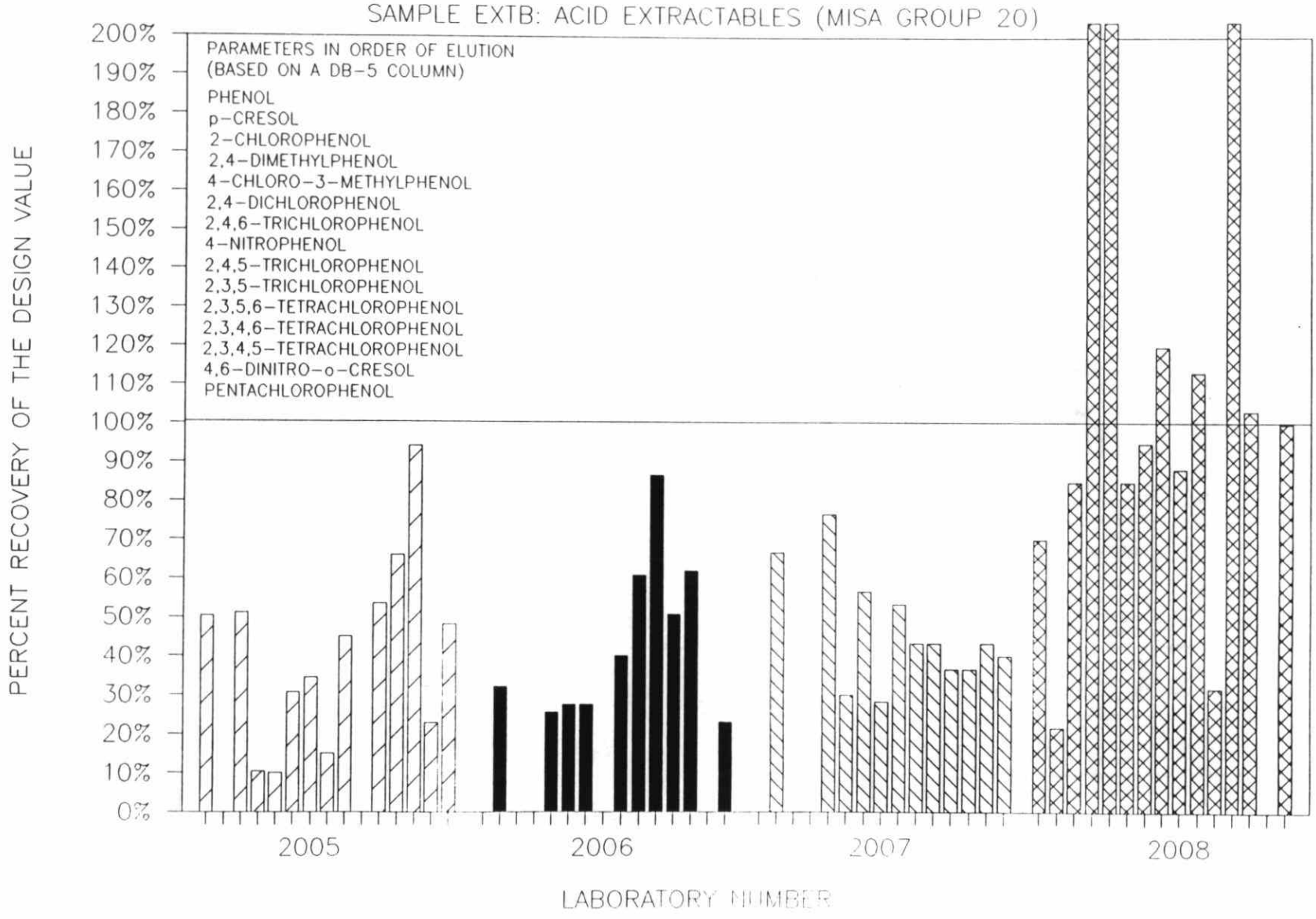
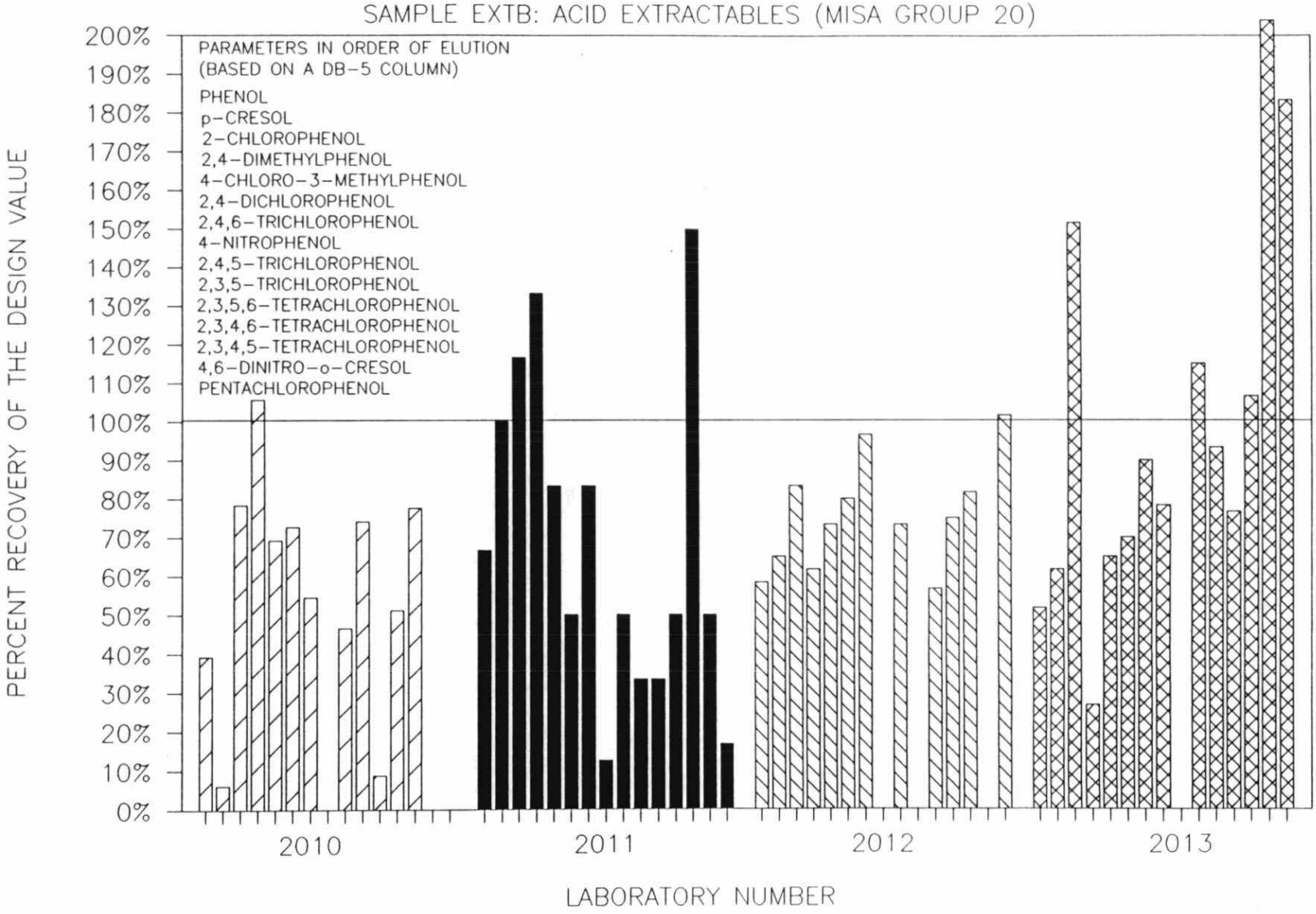


FIG. 23: INTERLABORATORY STUDY 89-5



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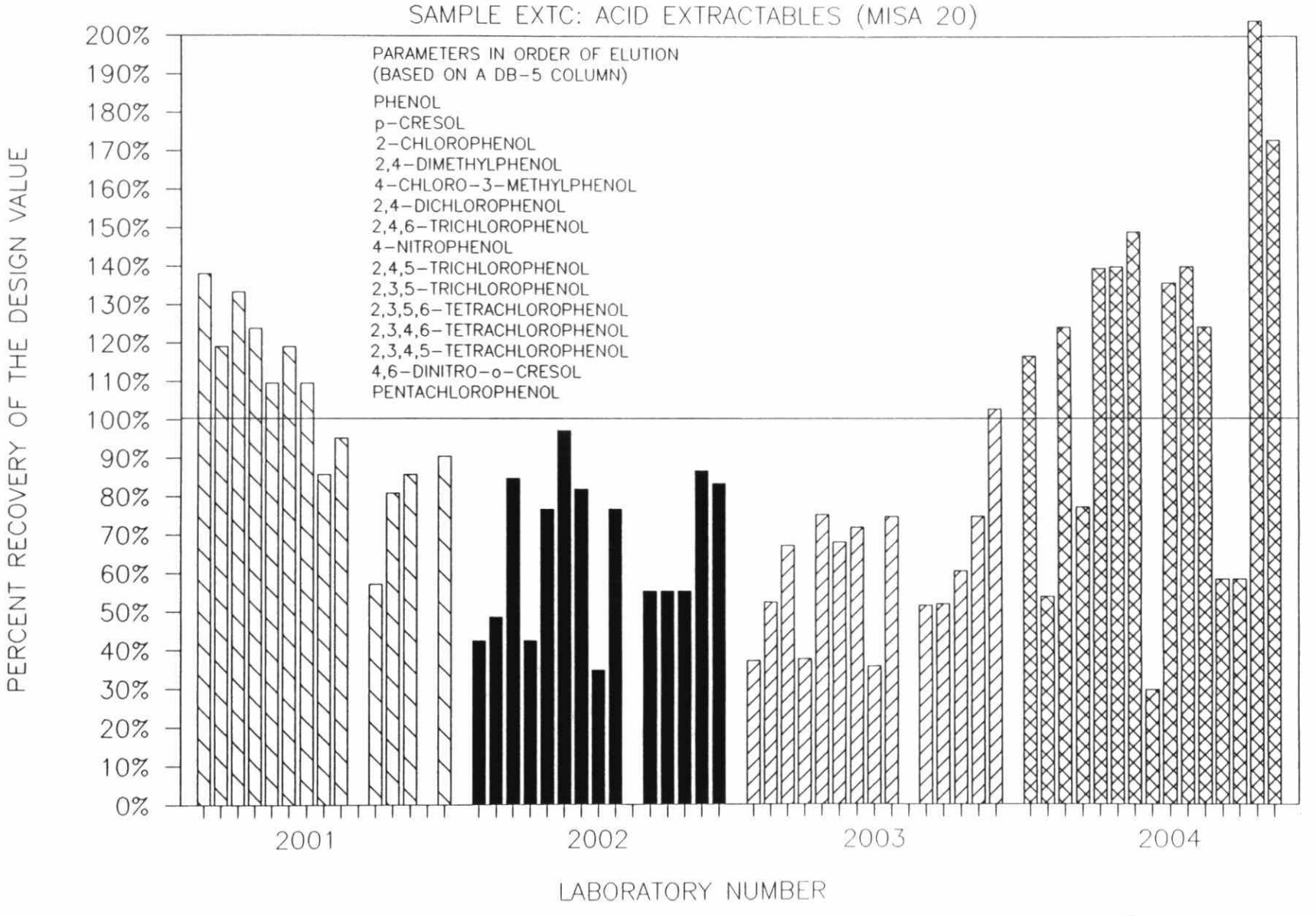

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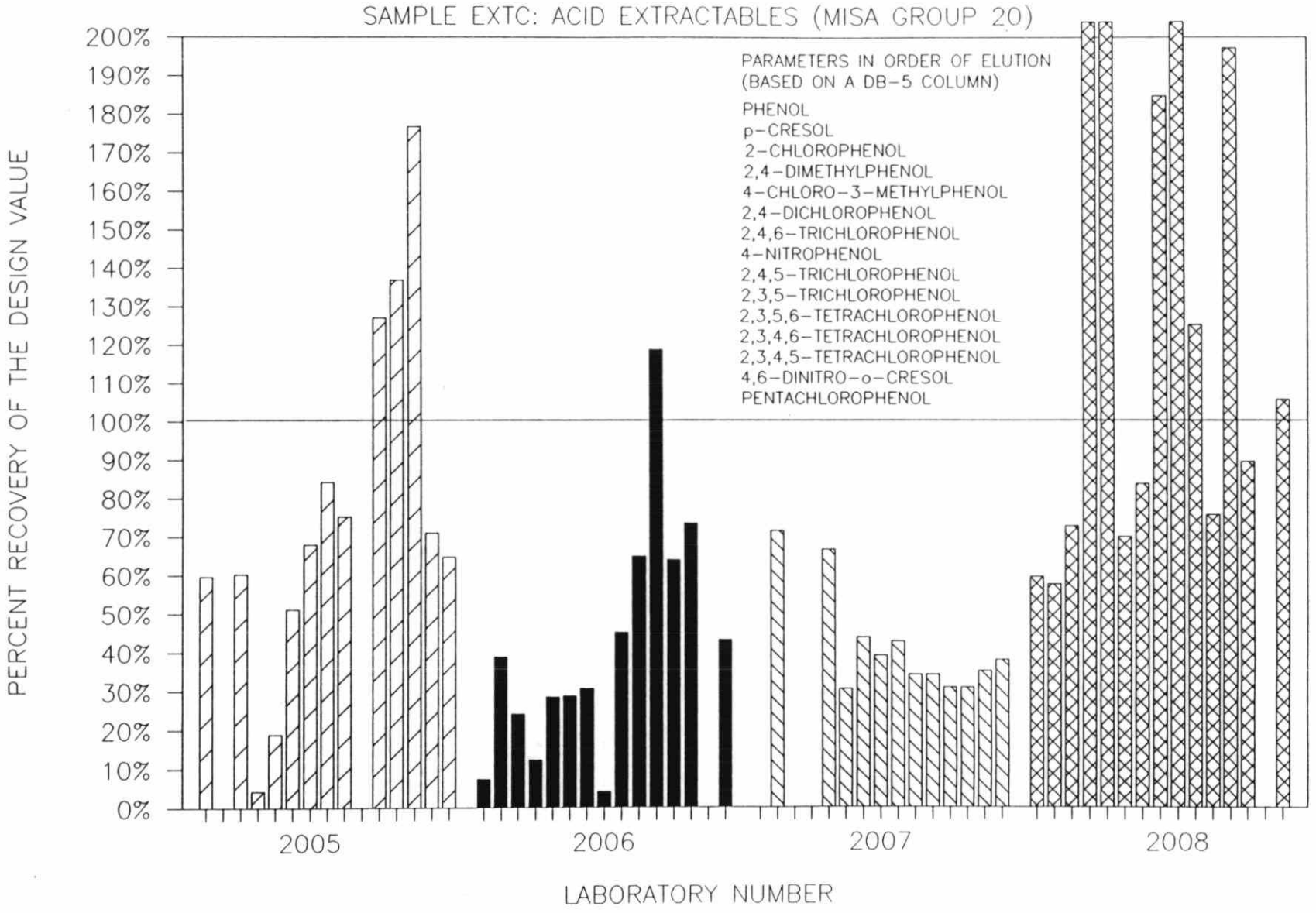

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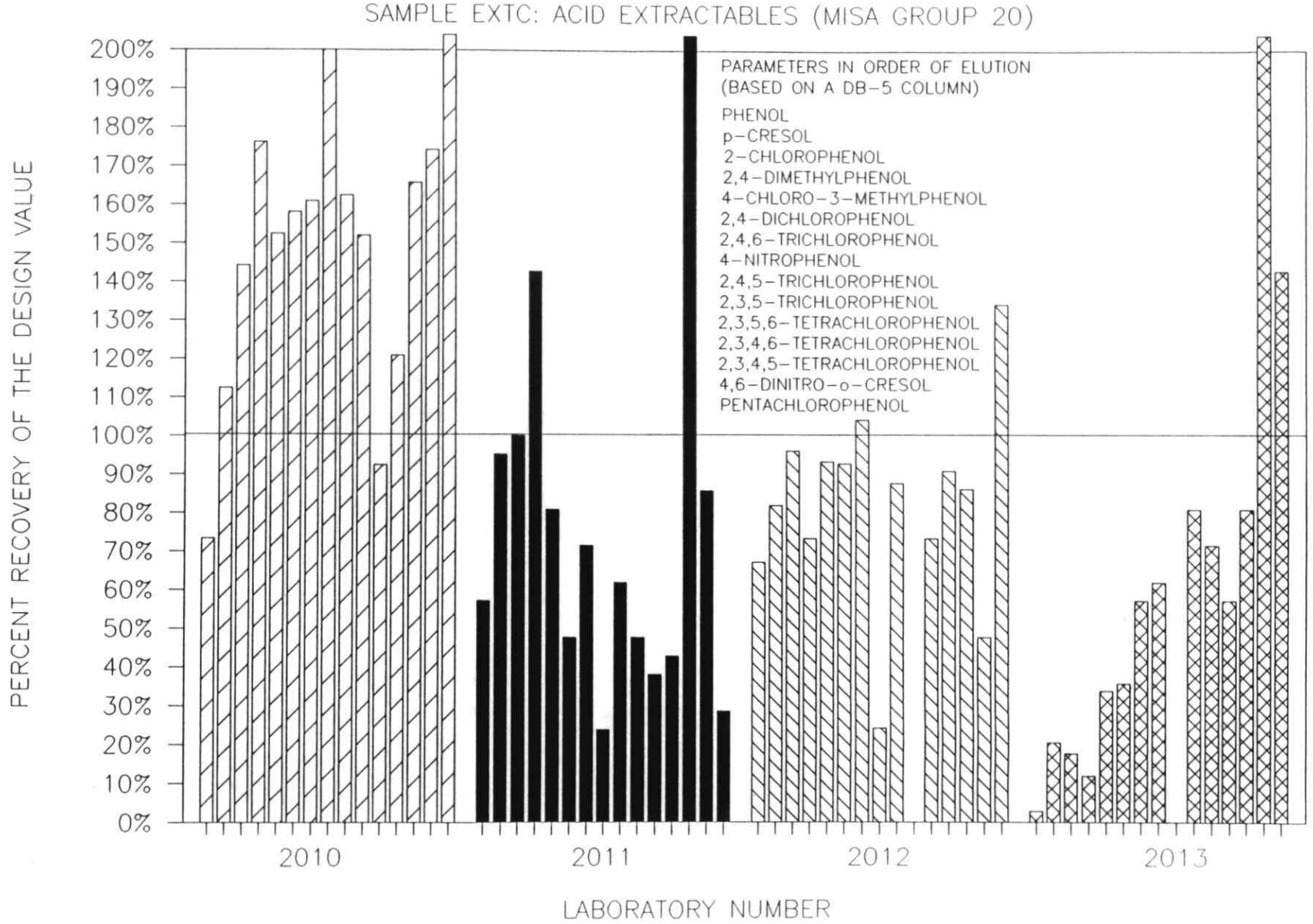

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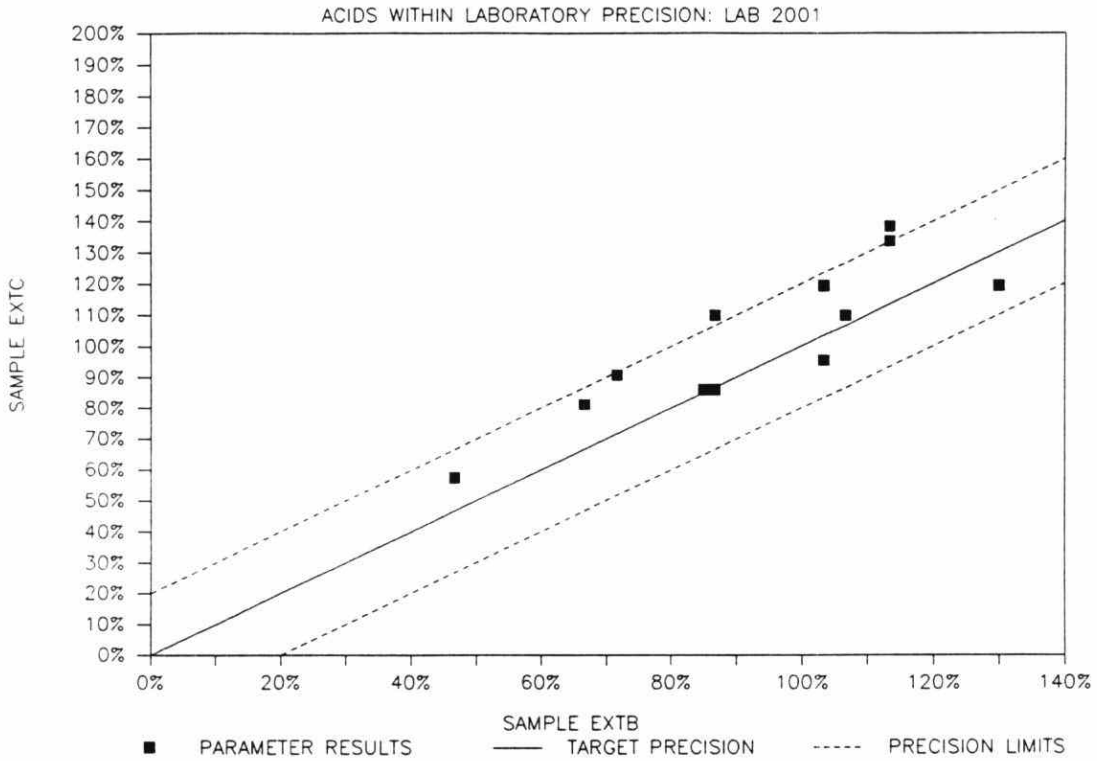

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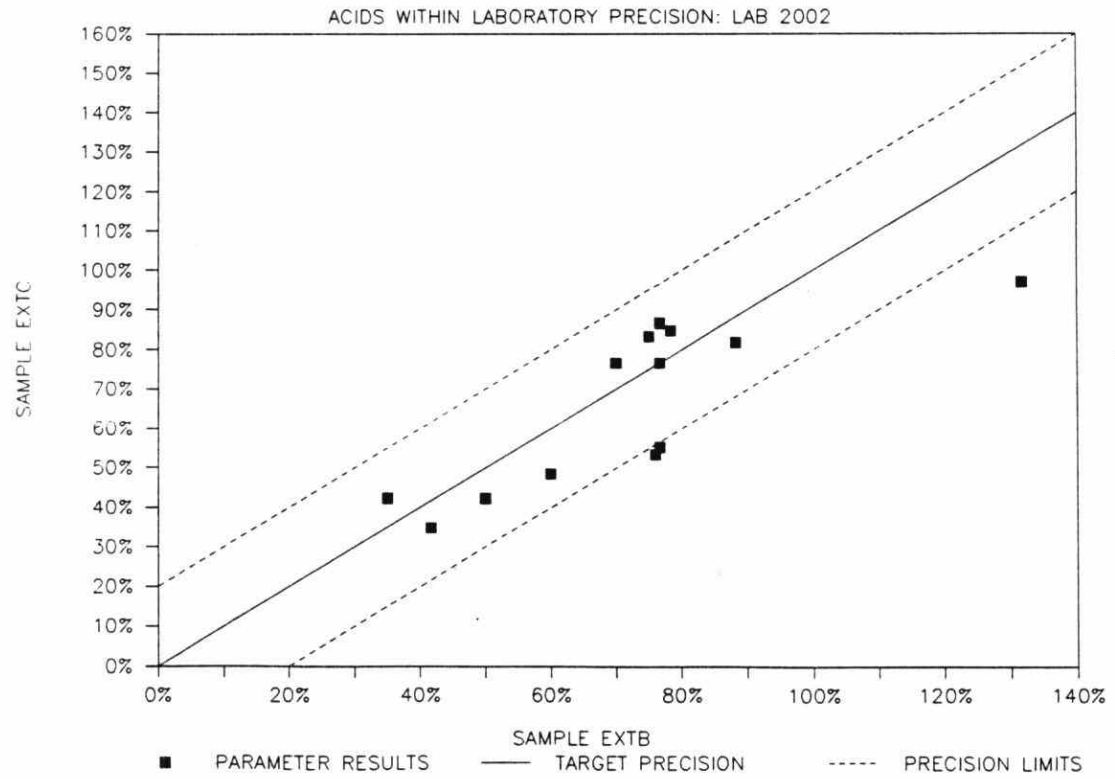

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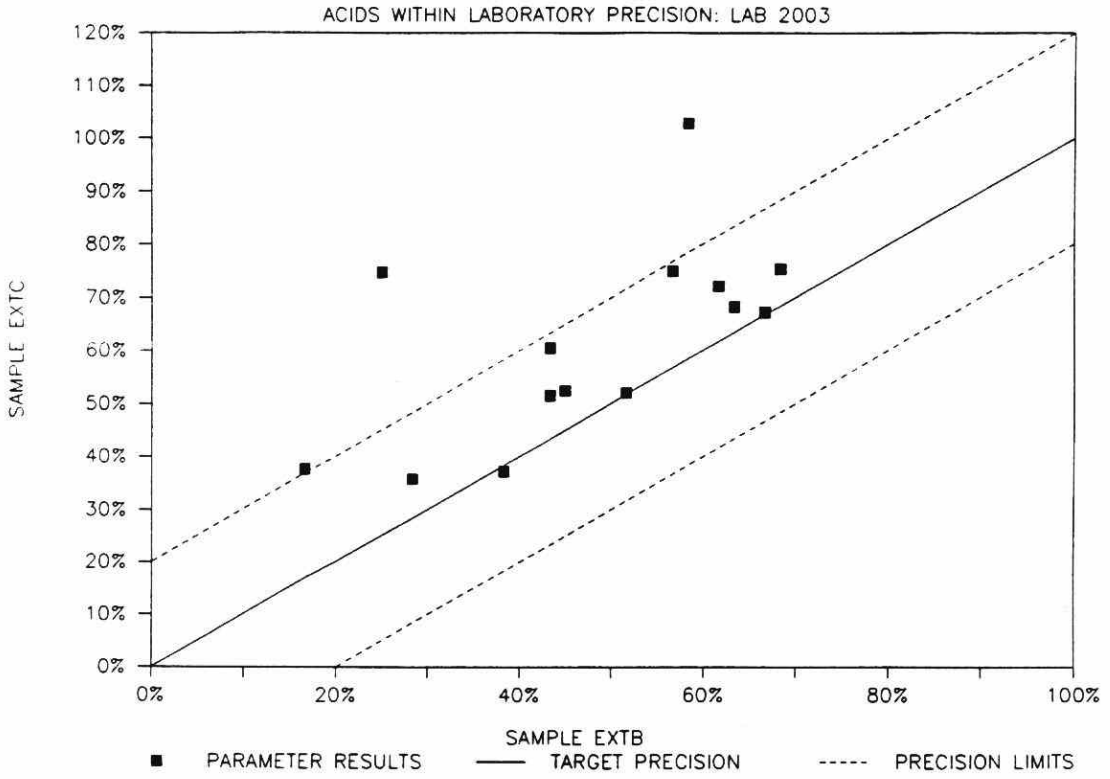

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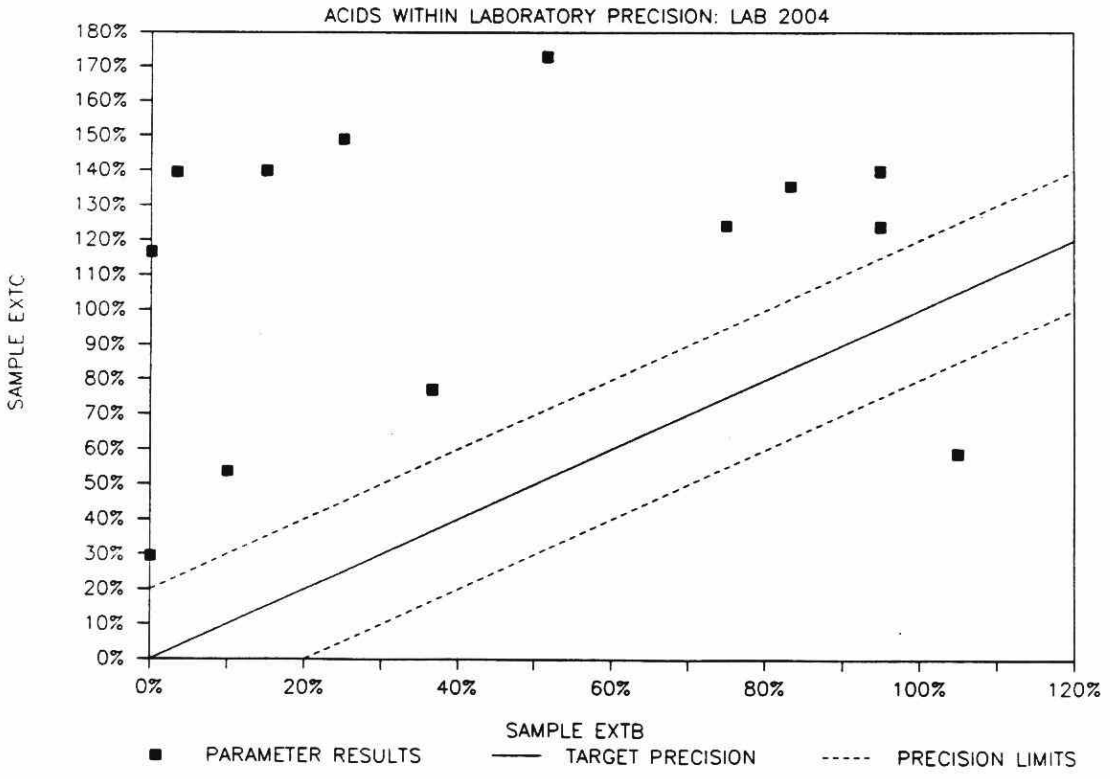

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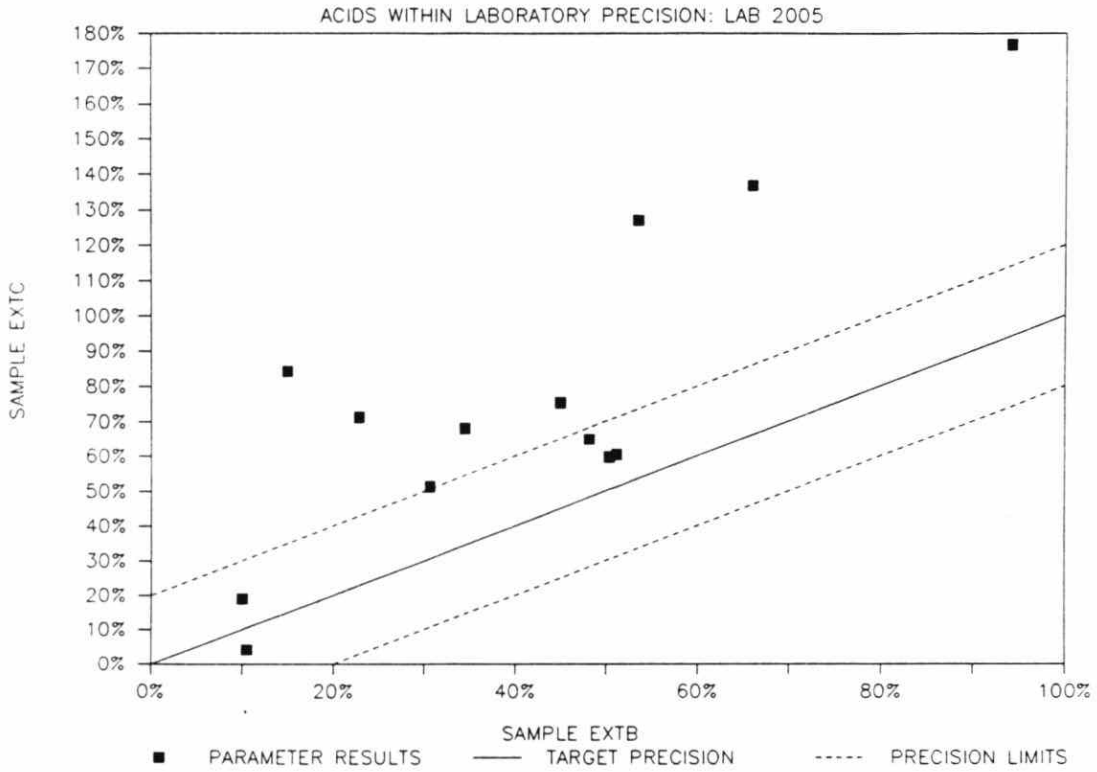

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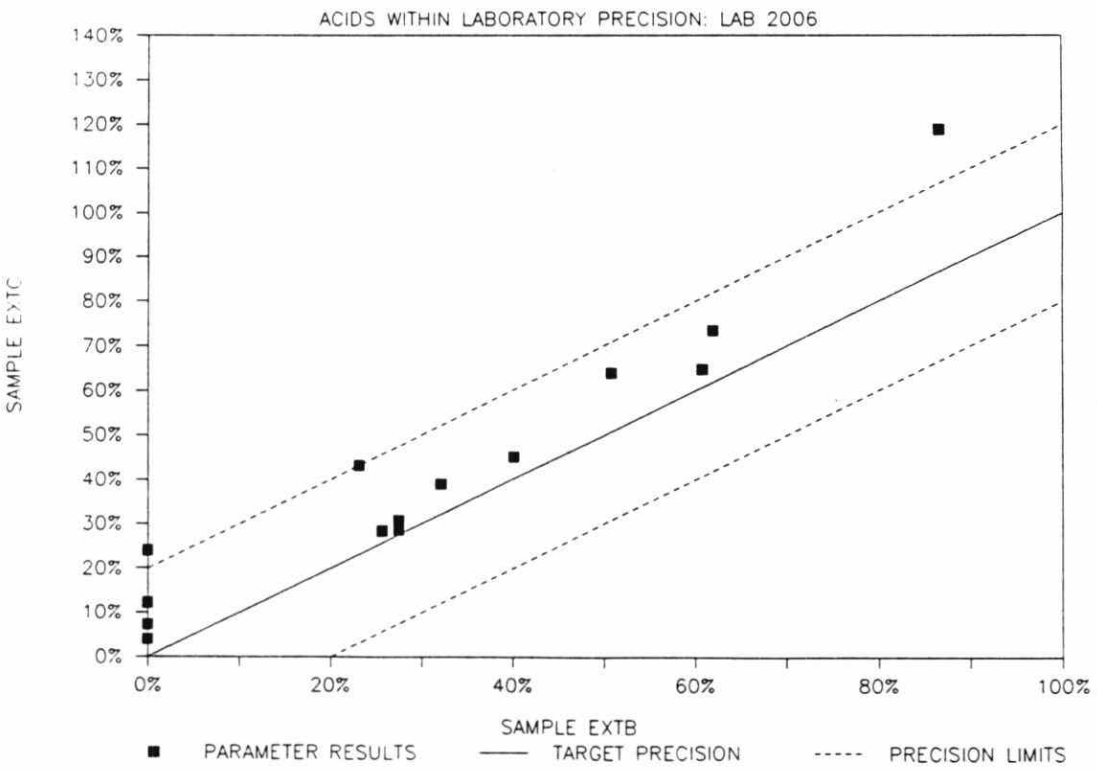

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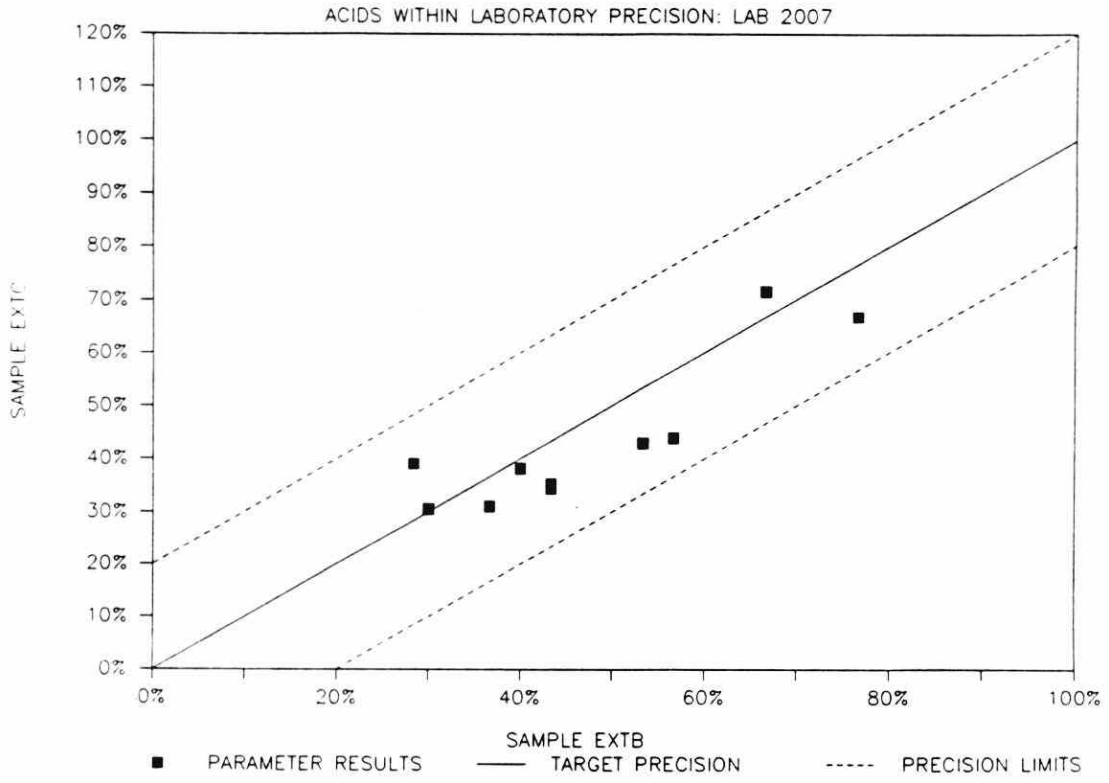

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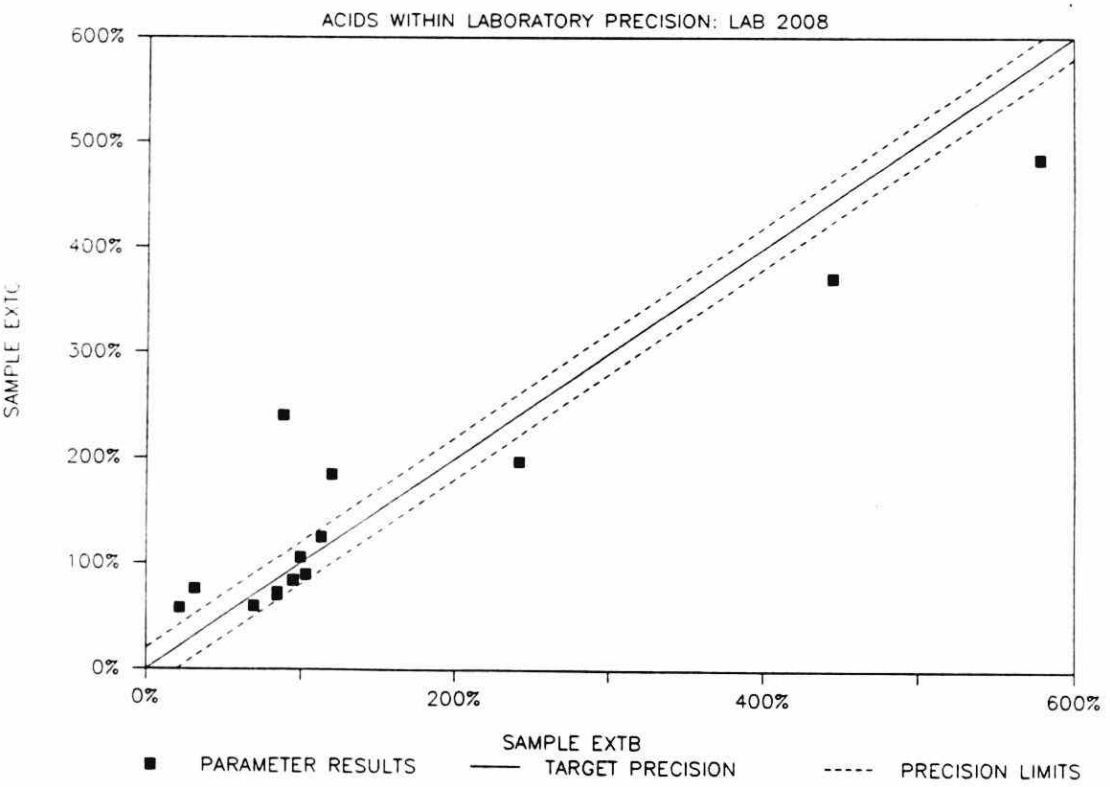

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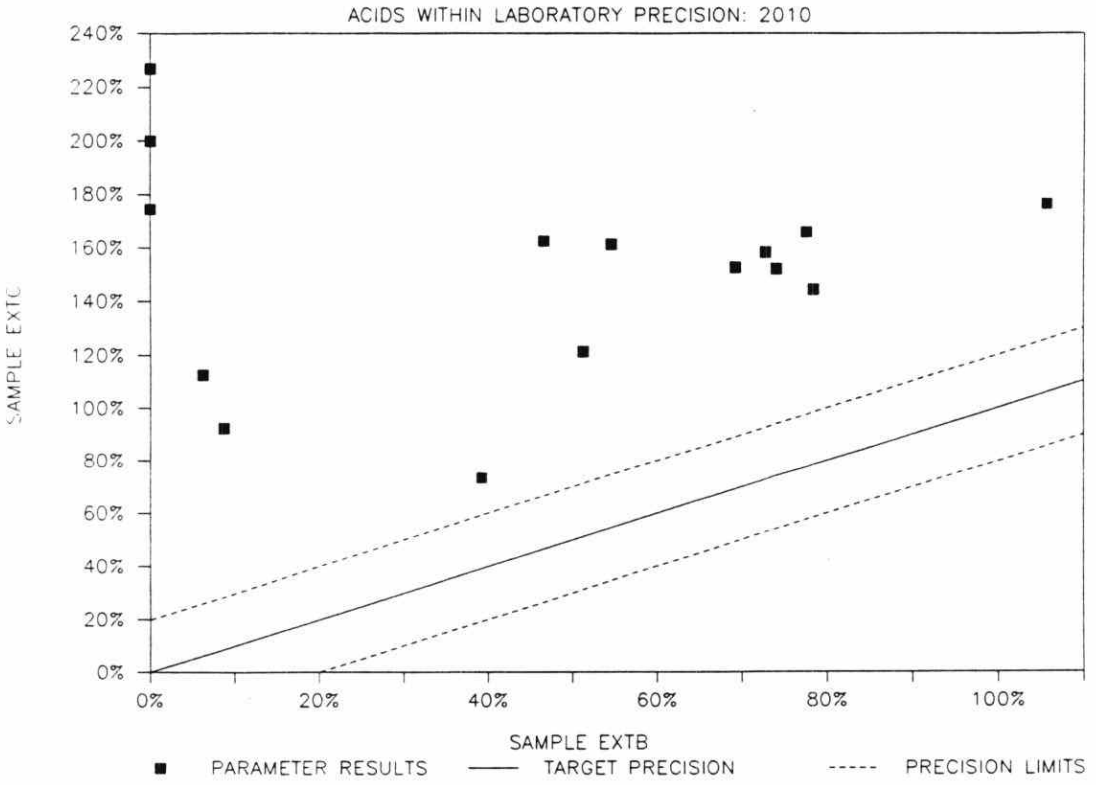

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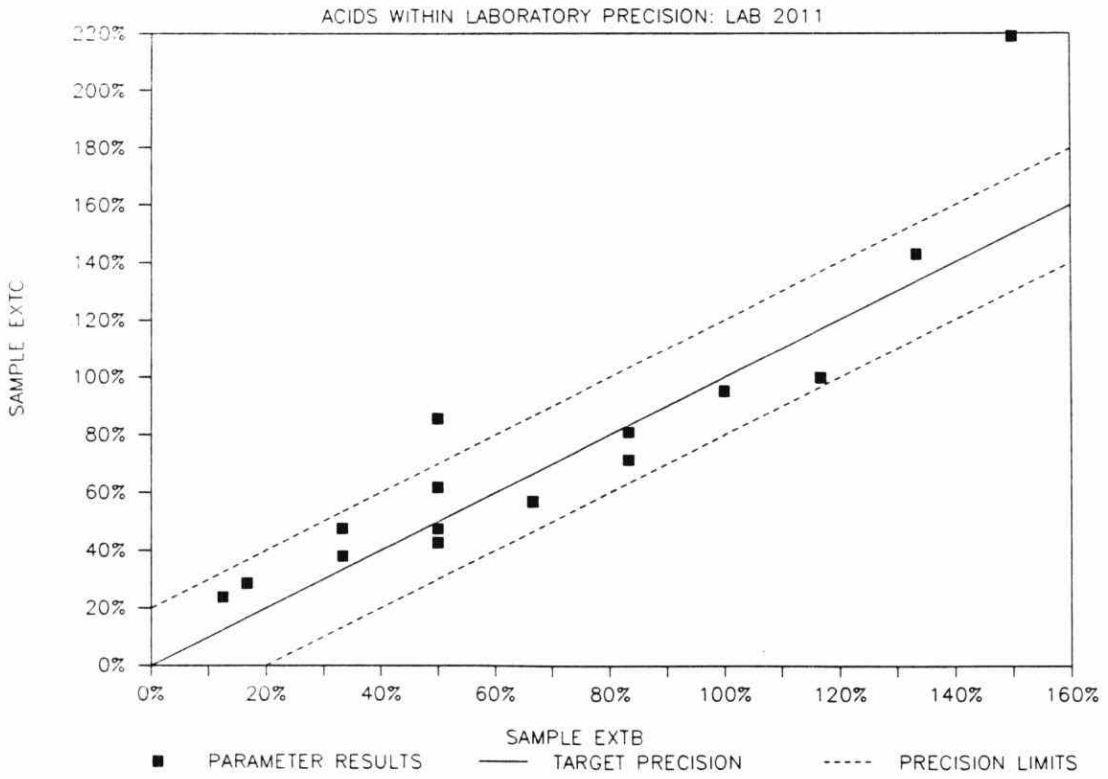

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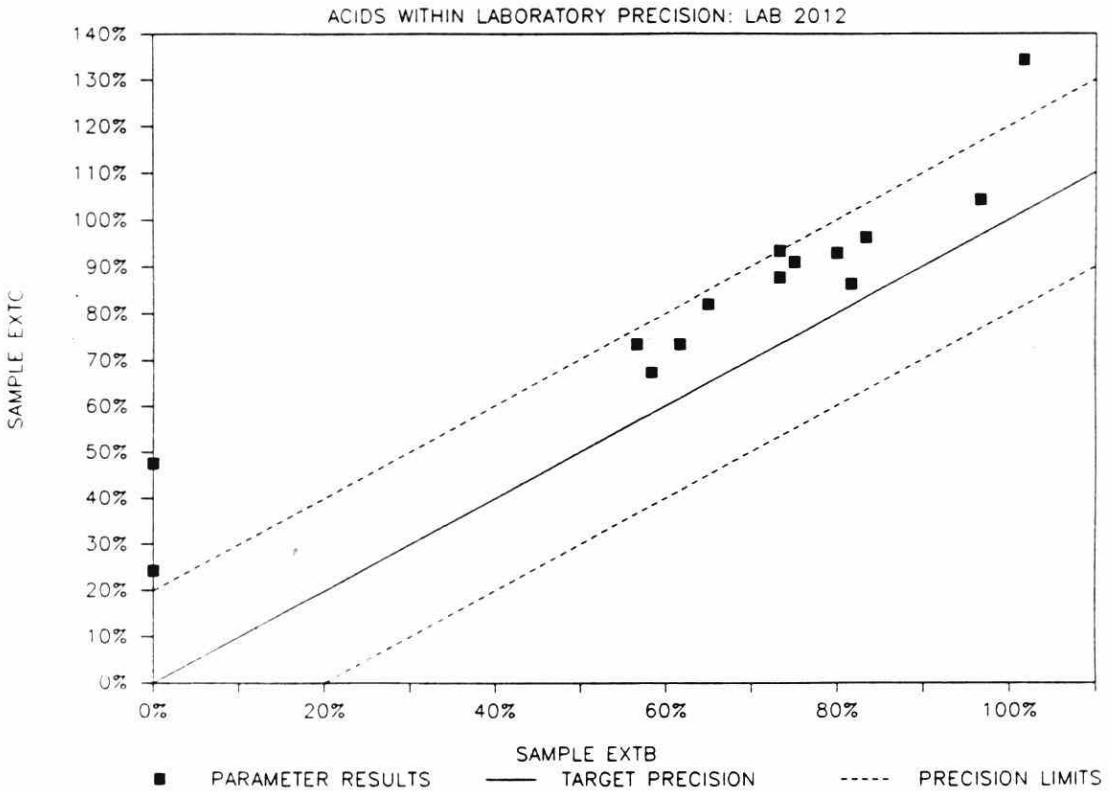

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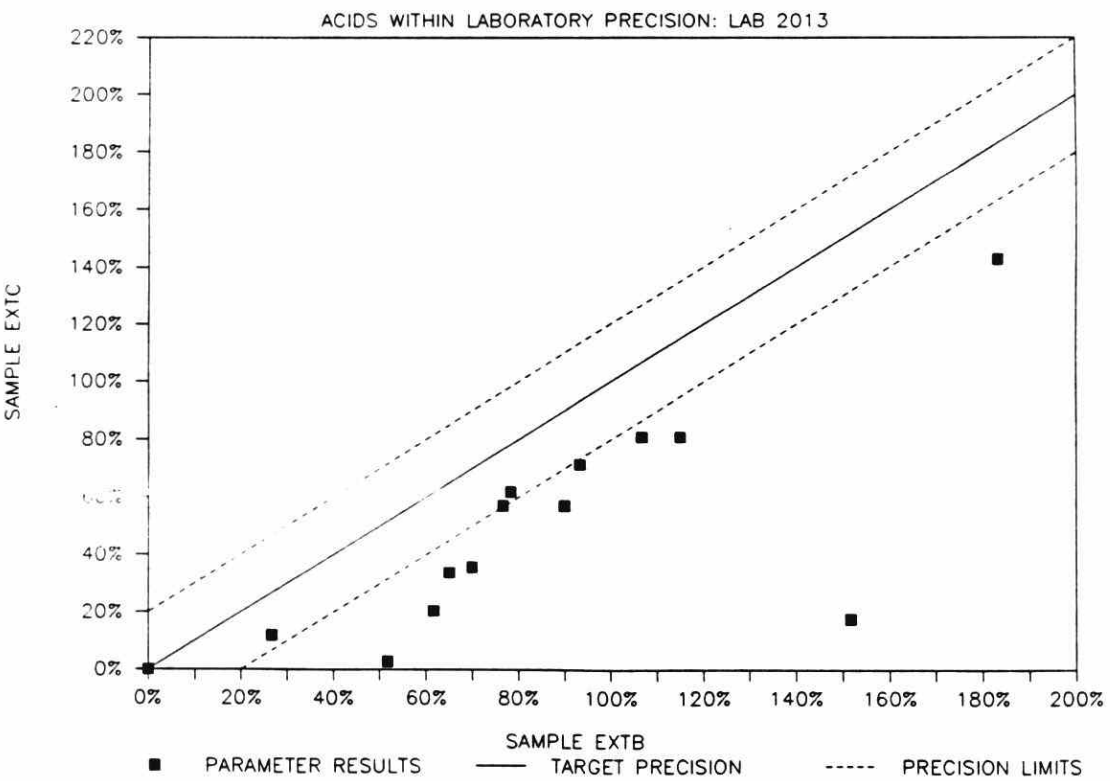

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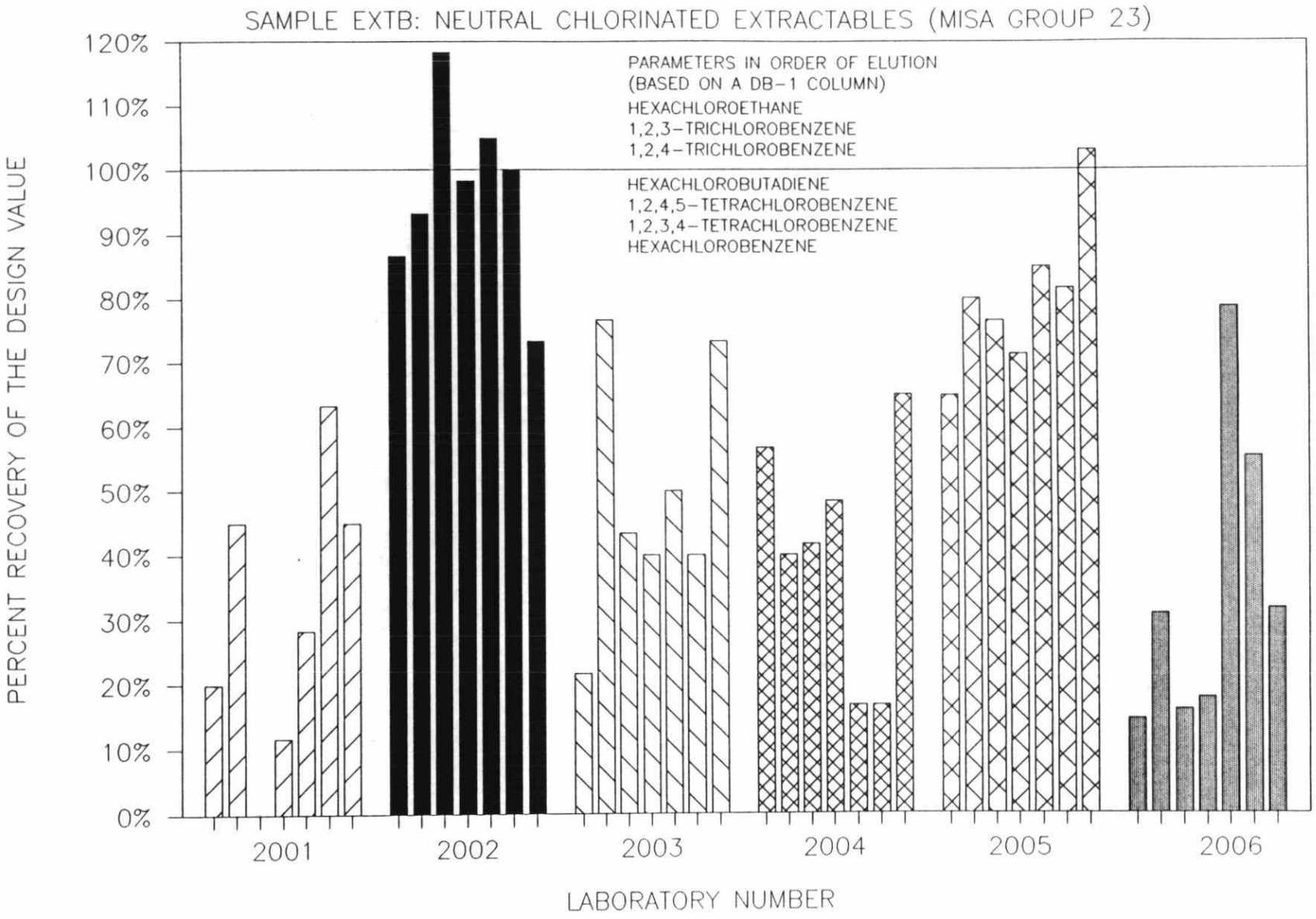

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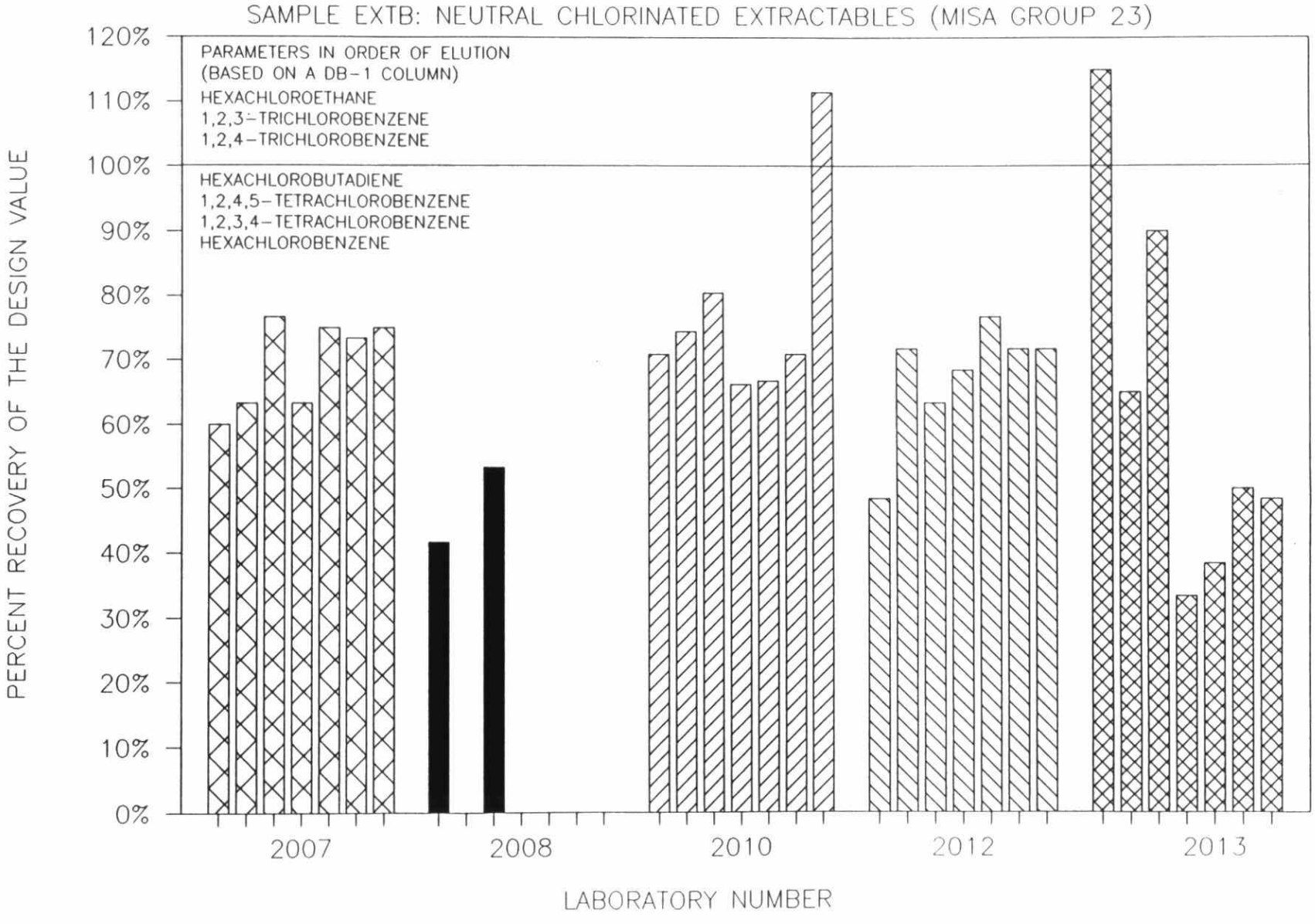

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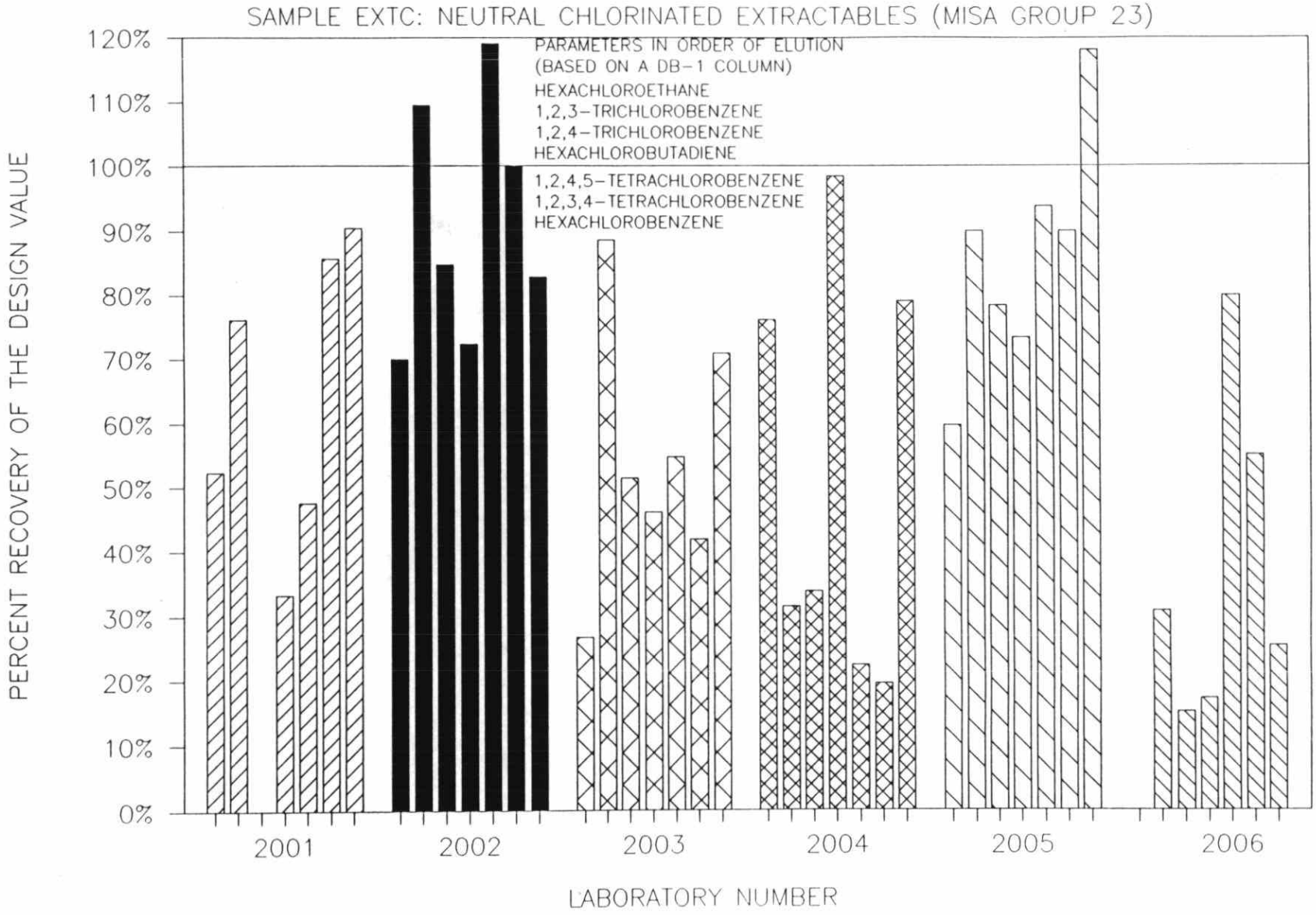

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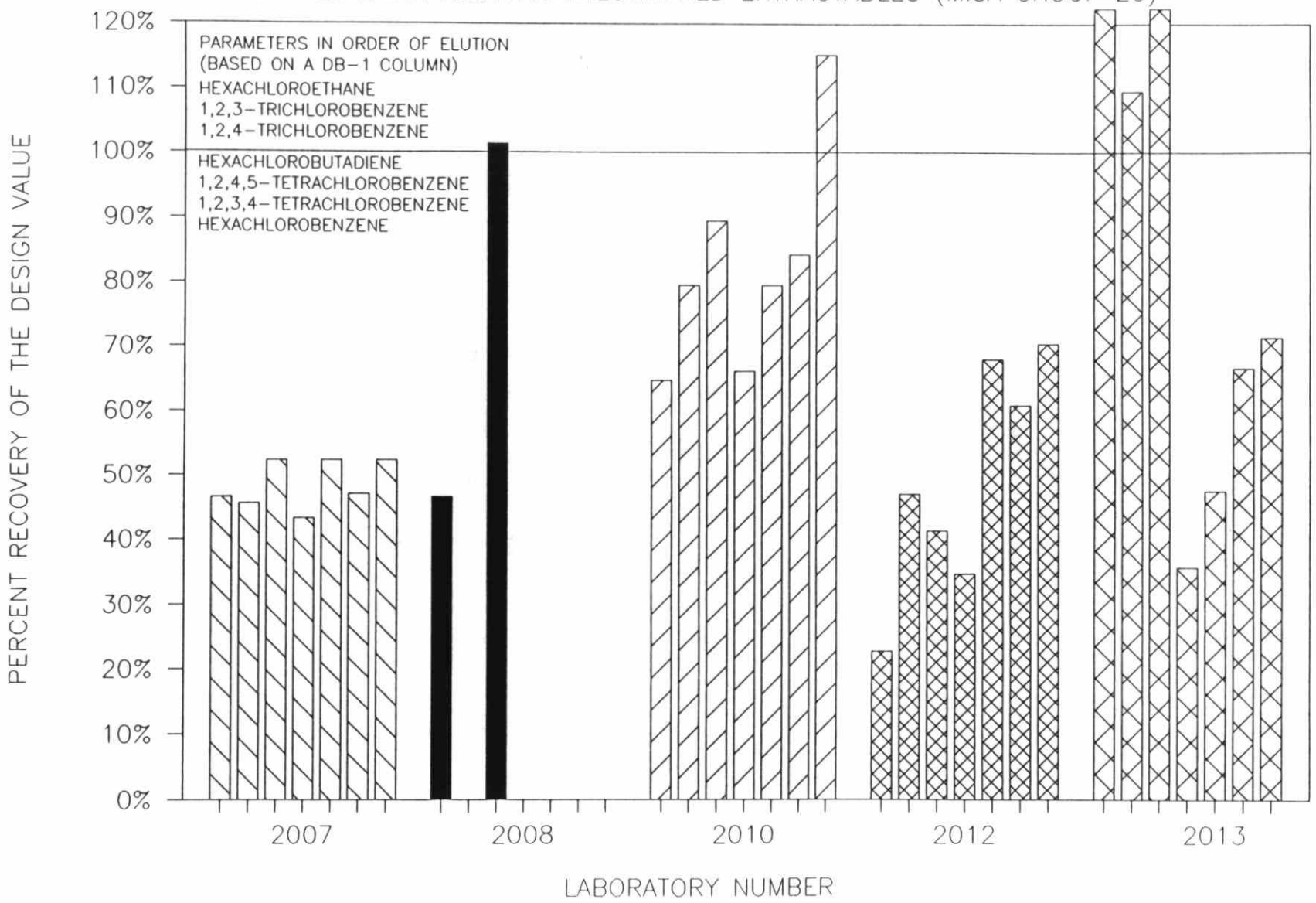

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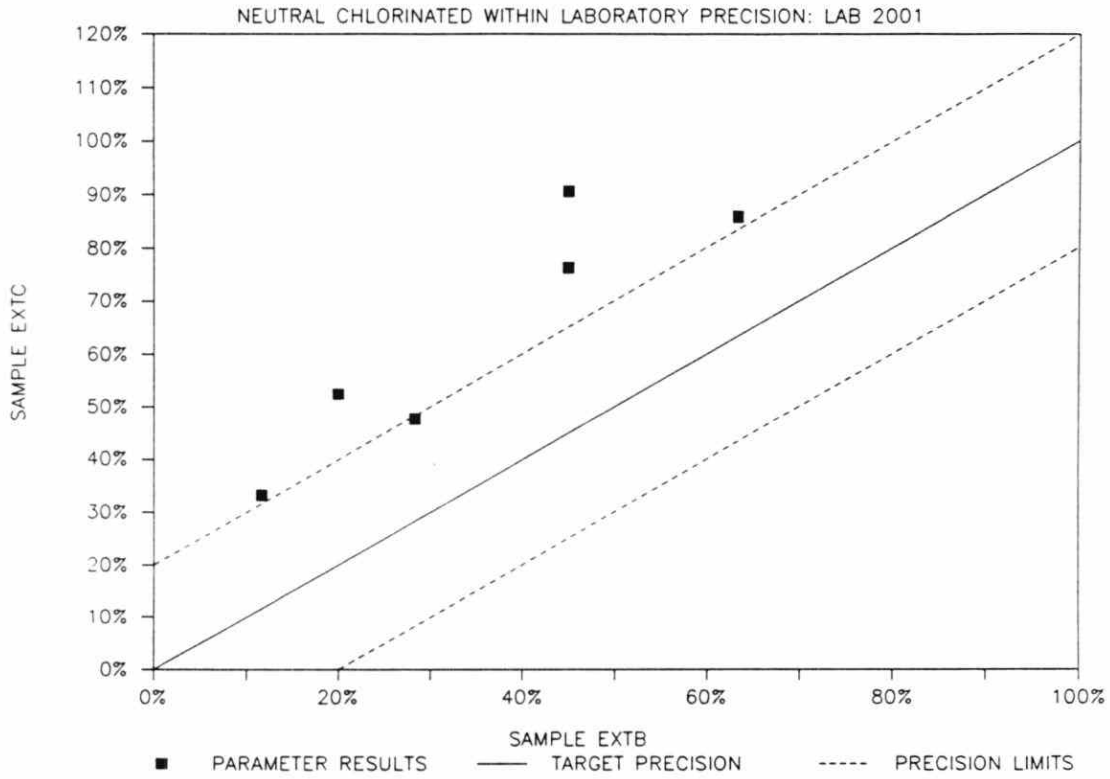
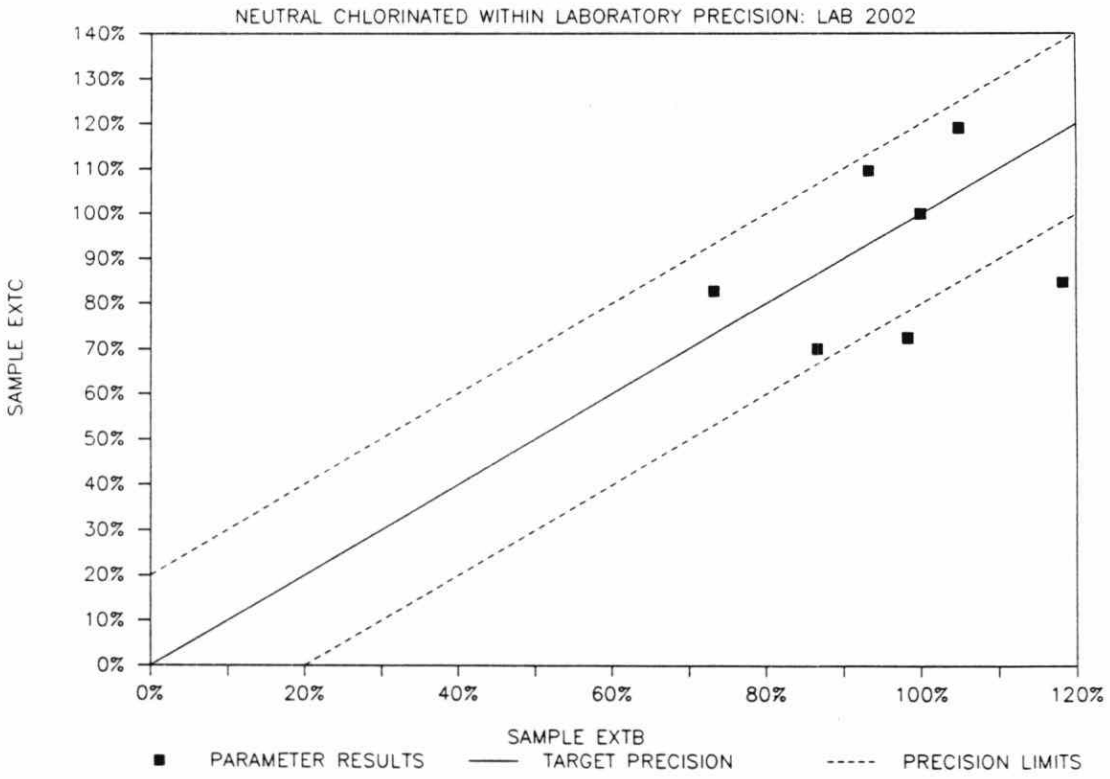


FIG. 44: INTERLABORATORY STUDY 89-5



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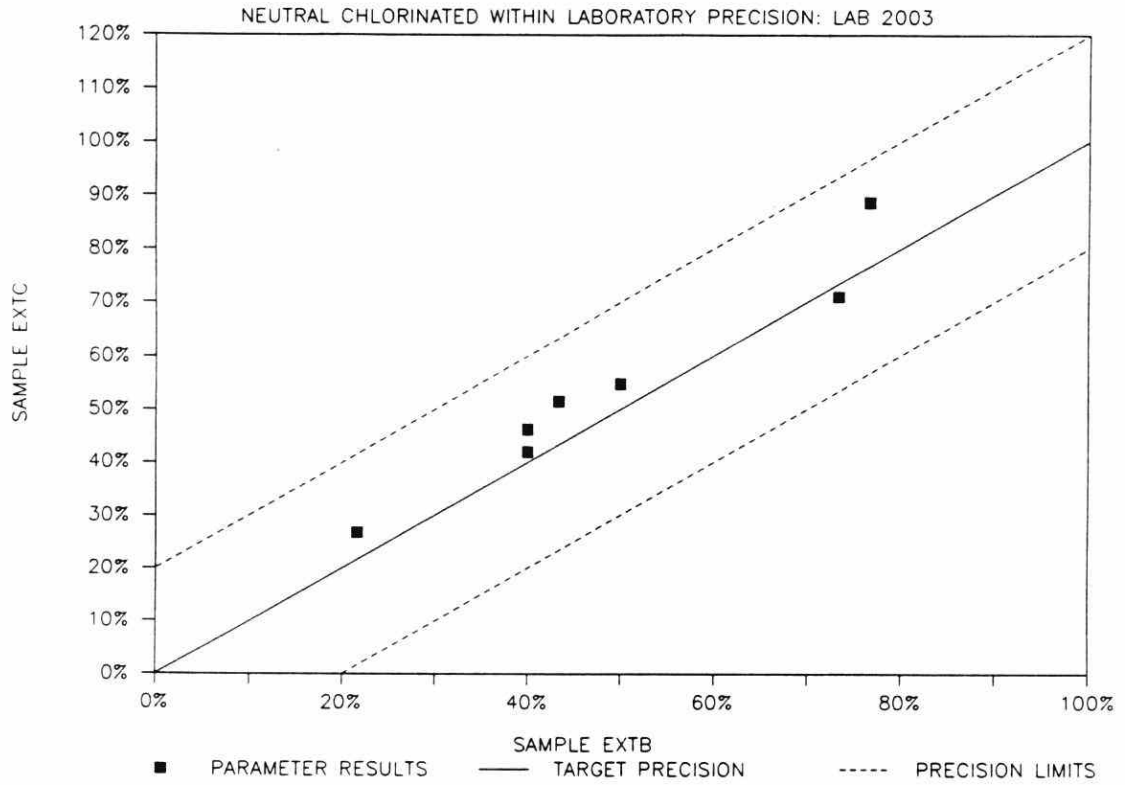

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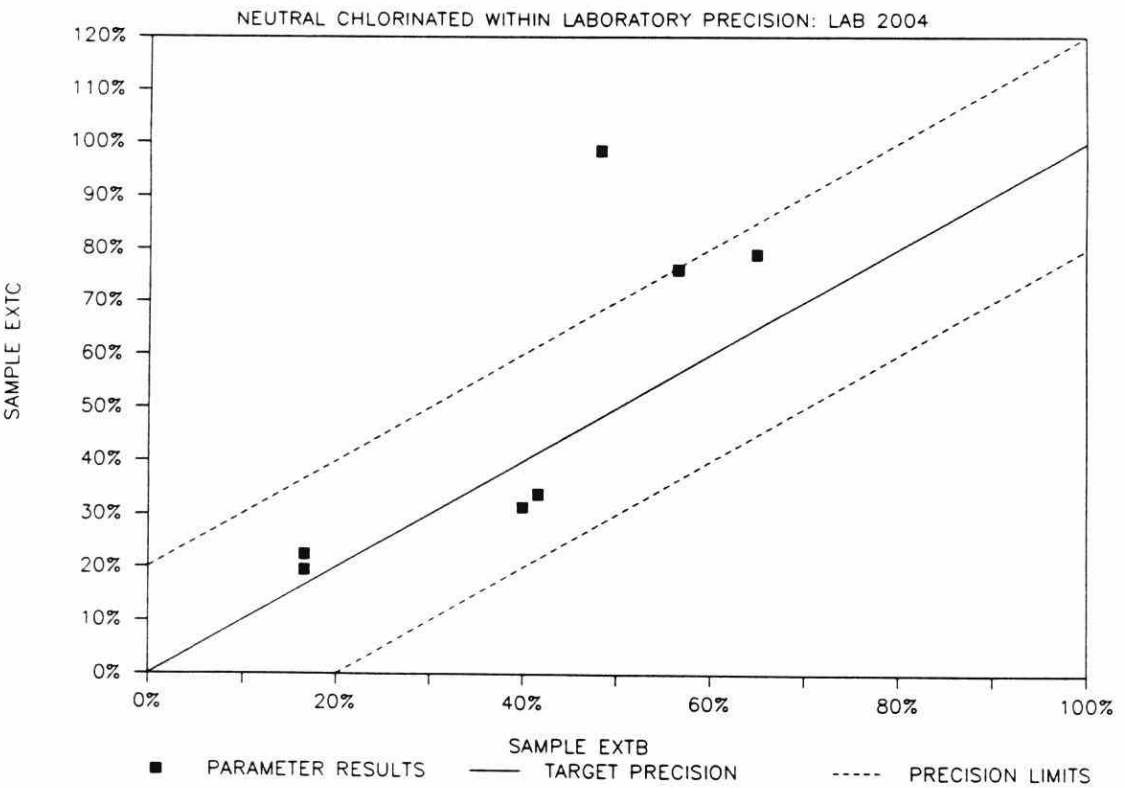
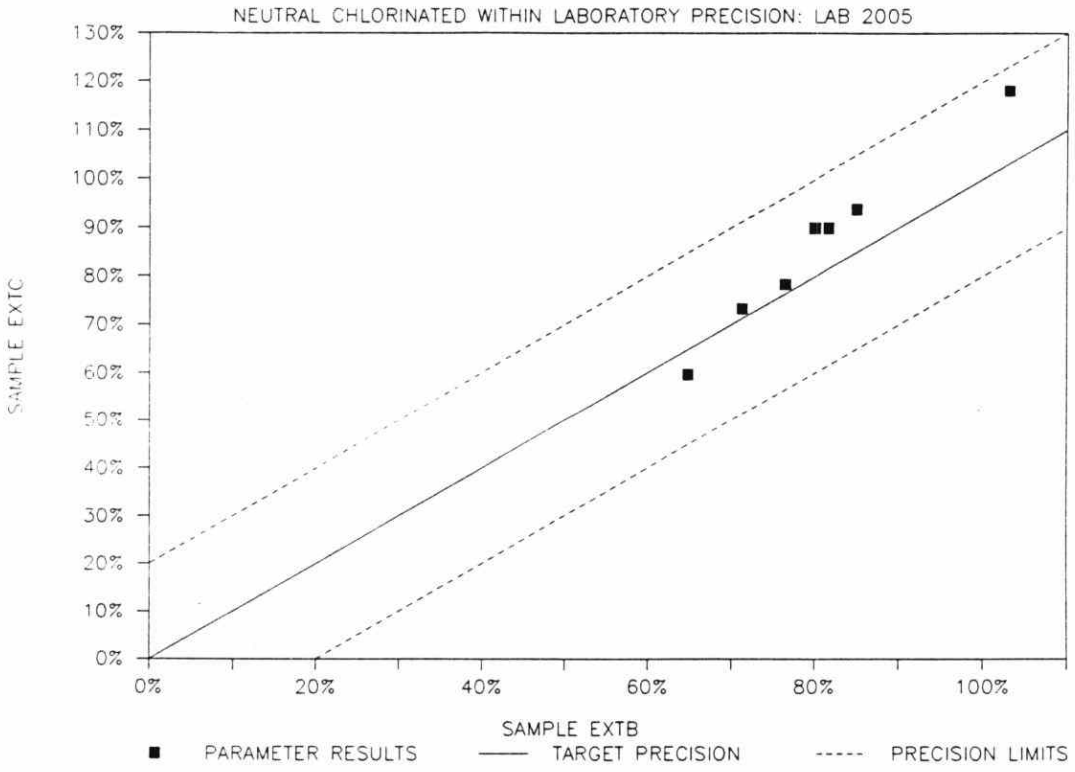


FIG. 47: INTERLABORATORY STUDY 89-5



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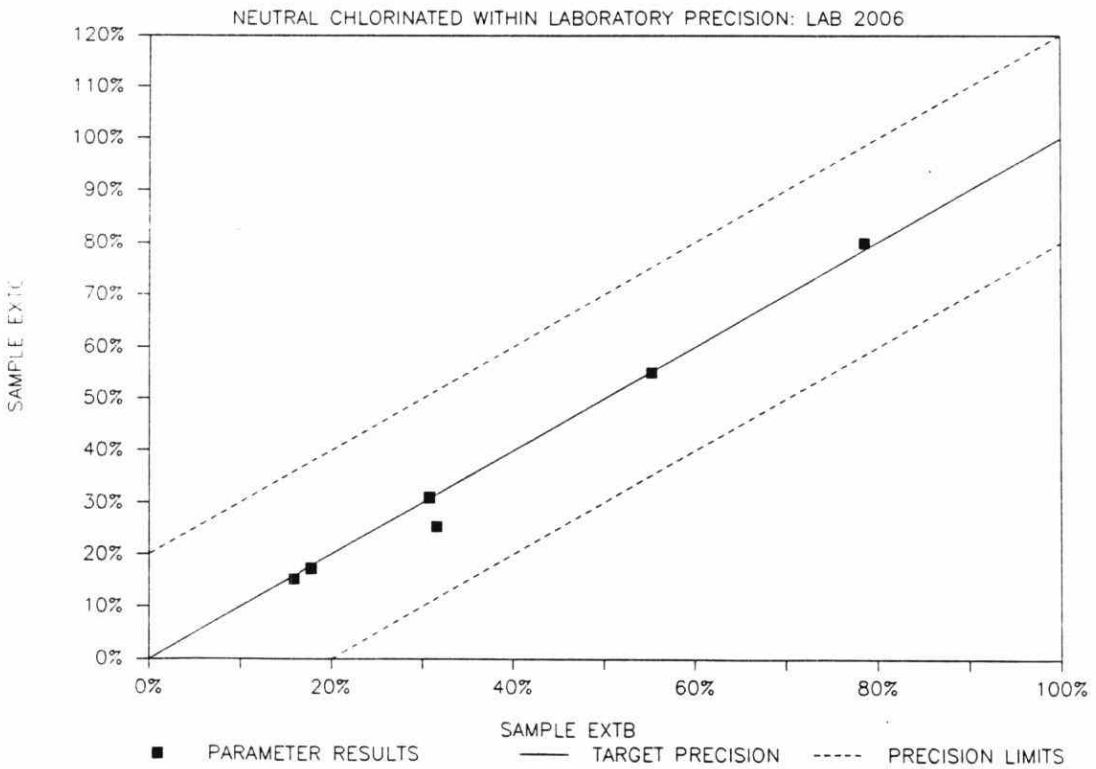

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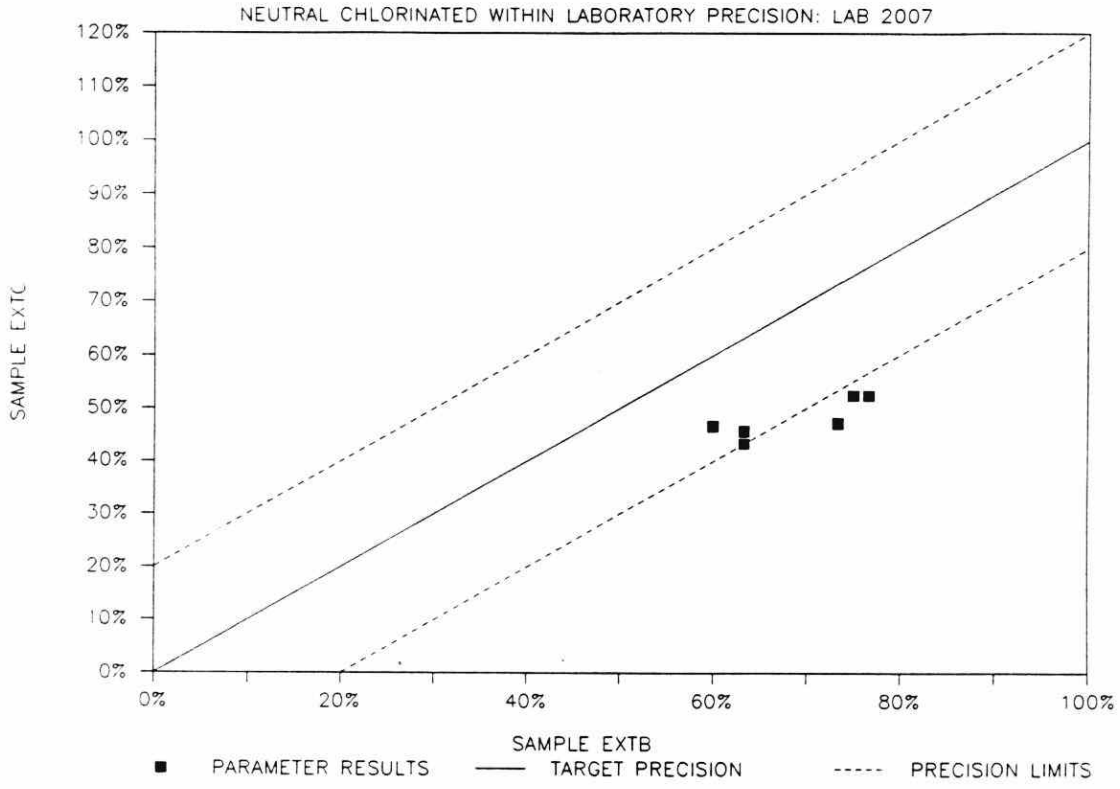

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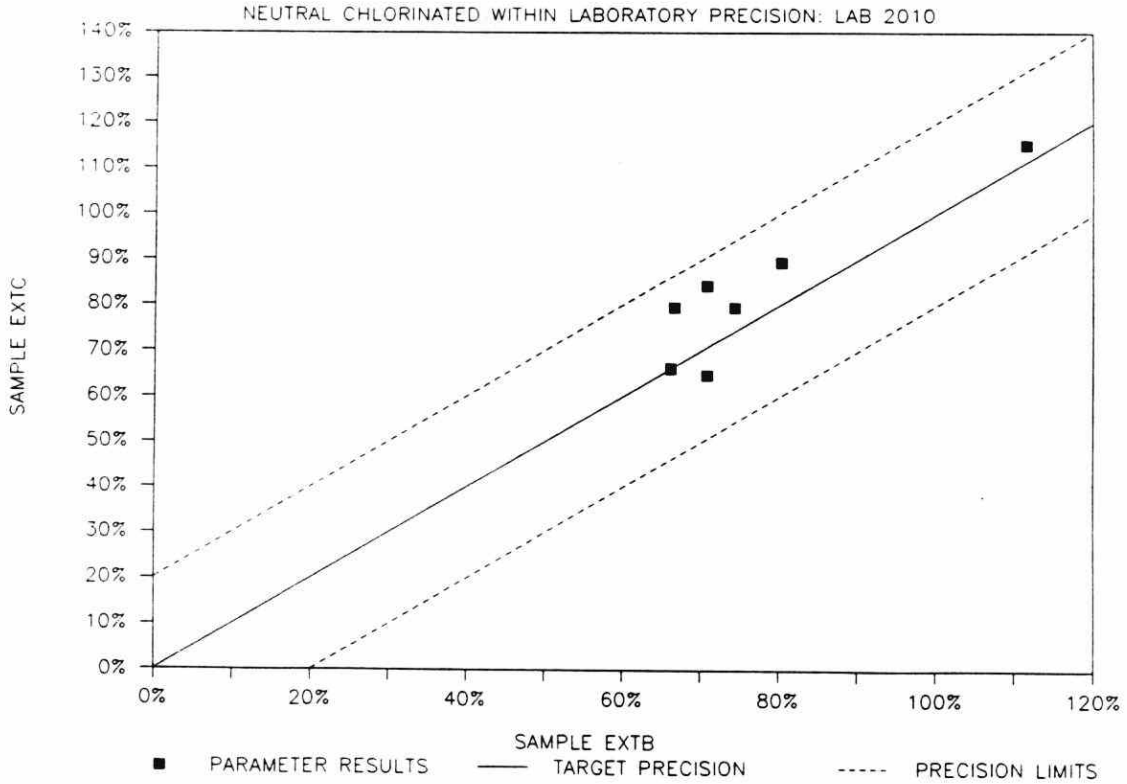

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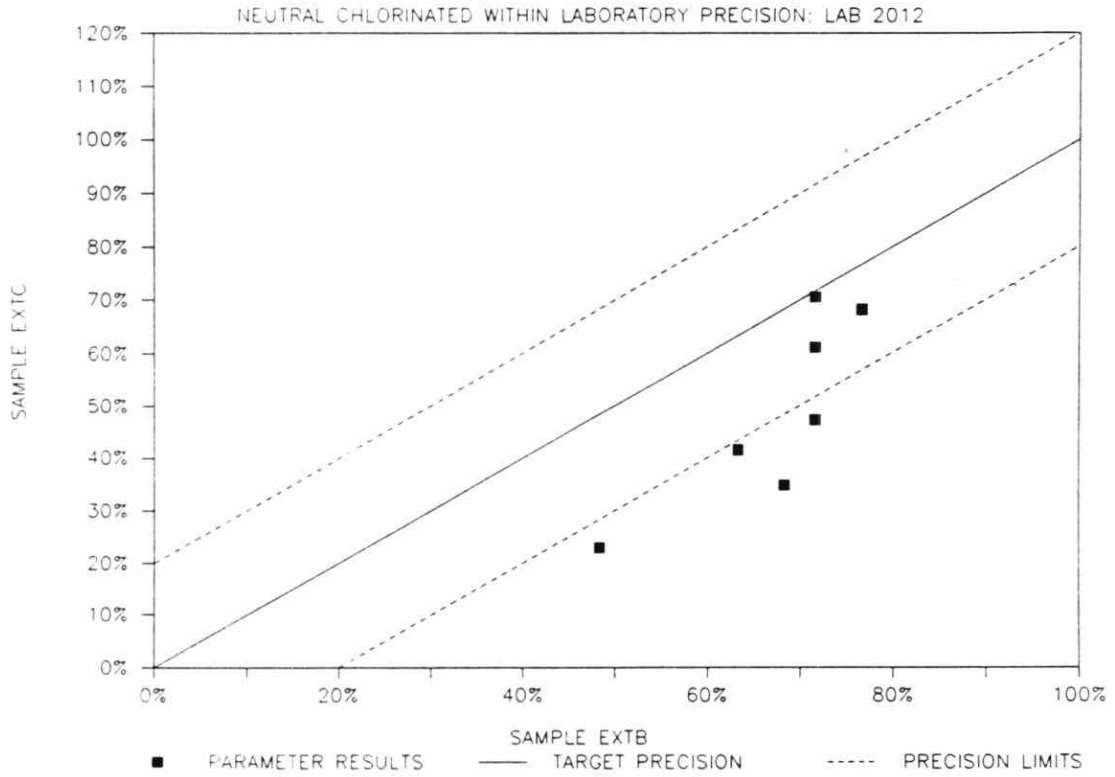

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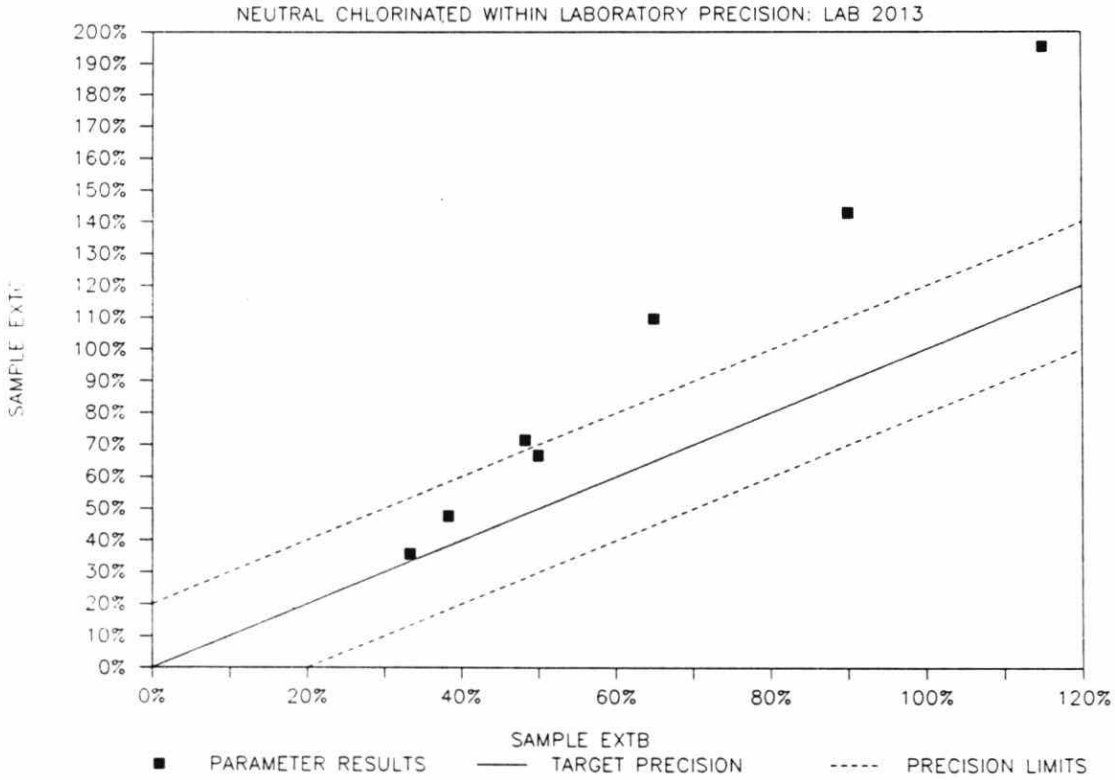

8 APPENDIX 2 - LIST OF PARTICIPANTS AND CORRESPONDENCE

Enviroclean
Environmental Laboratory Services
921 Leathorne St.
London, Ont., N5Z 3M7
(519) 686-7558

Contact: J. M. Latella

Ortech International
2395 Speakman Dr.
Mississauga, Ont., L5K 1B3
822-4111

Contact: Jack Brady

Barringer Laboratories
5735 McAdam Rd.
Mississauga, Ont., L4Z 1N9
890-8566

Contact: Alan Lipski

Clayton Environmental Consultants
949 McDougall Ave.
Windsor, Ont., N9A 1L9
(519) 255-9797

Contact: Paul Epstein

Walker Industries
P.O. Box 100
2800 Townline Rd.
Thorold, Ont., L2V 3Y8
(416) 227-4142

Contact: Douglas De Coppel

Microbe Inc.
Environmental Biotechnology
85 Midpark Rd.
London, Ont., N6N 1B2
(519) 668-1005

Contact: Shannon Flannigan/
Debbie Boersma

Eli-Eco Logic International
143 Dennis St.
Rockwood, Ont., N0B 2K0
(519) 856-9591
450-7691 (Toronto Line)

Contact: Elizabeth Chisholm

Zenon Environmental Inc.
5555 North Service Rd.
Burlington, Ont., L5L 5H7
(416) 332-8788

Contact: M. Glenys Foster

Wellington Environmental Consultants
389 Laird Rd.
Guelph, Ont. N1H 6J3
(519) 822-2436

Contact: Brock Chittim

Mann Testing Laboratories
5550 McAdam Rd.
Mississauga, Ont., L4Z 1P1
890-2555

Contact: Jim Forrester

Novalab
9420 Côte de Liesse
Lachine, Que., H8T 1A1
(514) 636-6218

Contact: Dr. John Fenwick

Canviro Analytical
Laboratories Ltd.
50 Bathurst Dr., Unit 12
Waterloo, Ont., N2V 2C5
(519) 747-2575

Contact: Dale Sutherland

Ontario Ministry of the Environment
Laboratory Services Branch
Trace Organics Section
125 Resources Rd.
Rexdale, Ont., M9W 5L1
(416) 235-5857

Contact: Yvonne Jones/
George Crawford

Beak Analytical Services
14 Abacus Rd.
Brampton, Ont., L6T 5B7
458-4044

Contact: John Robertson/Daniel Andrews

Dow Chemical Co.
Vidal St.
P.O. Box 3030
Sarnia, Ont., N7T 7M1
(519) 339-3264

Contact: Brian Worthington

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYIS OF TRACE ORGANIC COMPOUNDS

STUDY NO. 89-5

INTRODUCTION

Private laboratories receiving this notification are invited by the Ontario Ministry of the Environment to participate in an interlaboratory variability study of spiked reagent water conducted using MISA analysis protocols. Laboratories interested in participating in this program, scheduled for the week of May 1, 1989, should contact Sylvia Cussion at (416) 235-5842 or Catherine Doehler (416) 235-6055 of the Ministry of the Environment to confirm participation no later than April 25, 1989. All participants should follow-up telephone acceptance with written confirmation (FAX - (416) 235-5744) by April 28, 1989.

BACKGROUND

This study is being conducted to assist laboratories in assessing their analytical performance. All procedures should follow those principles and protocols outlined in the MISA regulations (Ontario Reg. 358/88). Sample sets will include three samples per scan: a blank, a low spike (approx. 5 times the MDL) and a high spike (approx. 20 times the MDL).

NOTE: Any laboratory that does not have a copy of the MISA general regulations should contact Catherine Doehler for additional information.

The following scans are to be included in this round robin:

Extractables (to be analyzed by GC/MS)
(MISA Groups 19,20, and 23)

Time Limit: 30 days storage

Acenaphthene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(g,h,i)perylene
2-Chloronaphthalene
Chrysene
Dibenzo(a,h)anthracene
Fluoranthene
Fluorene
Naphthalene
Phenanthrene
Pyrene
Benzylbutylphthalate
Bis(2-ethylhexyl)phthalate
Di-n-butylphthalate
4-Bromophenyl phenyl ether

2,3,4,5-Tetrachlorophenol
2,3,4,6-Tetrachlorophenol
2,3,5,6-Tetrachlorophenol
2,3,5-Trichlorophenol
2,4,5-Trichlorophenol
2,4,6-Trichlorophenol
2,4-Dimethylphenol
2,4-Dichlorophenol
4,6-Dinitro-o-cresol
2-Chlorophenol
4-Chloro-3-methylphenol
4-Nitrophenol
p-Cresol
Pentachlorophenol
Phenol

1,2,3,4-Tetrachlorobenzene

4-Chlorophenyl phenyl ether	1,2,4,5-Tetrachlorobenzene
Bis(2-chloroethyl)ether	1,2,3-Trichlorobenzene
2,4-Dinitrotoluene	1,2,4-Trichlorobenzene
2,6-Dinitrotoluene	Hexachlorobenzene
Bis(2-chloroethoxy)methane	Hexachlorobutadiene
N-Nitrosodi-n-propylamine	Hexachloroethane

SCHEDULE

During the week of May 1, 1989 participating laboratories will receive a total of three (3) samples for analysis of Acid and Base/Neutral Extractables. All samples will be spiked reagent water samples.

Participating laboratories are expected to analyze the samples within the time limits specified in Schedule 2 of the general MISA regulations (Ontario Reg. 358/88). Blank reporting forms will be provided with the samples. Results for all analyses are to be reported within forty (40) days of receipt of the samples to Sylvia Cussion/Catherine Doehler at the following address:

Ministry of the Environment
Laboratory Services Branch
Laboratory Computer Systems - QA/QC Section
125 Resources Rd., P.O. Box 213
Rexdale, Ontario
M9W 5L1

SUMMARY OF RESULTS

All participating laboratories will be assigned a unique identification code. All laboratories will receive a complete set of the results, including a ranking for each laboratory where they will be identified only by their identification code. Recommendations made by the MOE will also be provided to the individual labs. Results will remain confidential and will only be released with the written permission of the individual participants.

It is the intent of this round robin (along with others) to assess the interlaboratory variability and detection capability for a broad range of organics and inorganics.

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYIS OF TRACE ORGANIC COMPOUNDS

STUDY NO. 89-5

☐ Yes, we will participate in MOE Study 89-5

☐ No, we will not participate in MOE Study 89-5

For the completeness of our records, and to avoid any shipping delays, please fill in the following:

Mailing Address: Shipping Address:

_____ _____

_____ _____

_____ _____

_____ _____

_____ _____

_____ _____

Contact Person: _____

Phone Number: _____

Please return this questionnaire before April 28, 1989 to:

Sylvia Cussion/Catherine Doehler
Ontario Ministry of the Environment
Laboratory Services Branch
LCS-QA/QC Section
125 Resources Rd.
Rexdale, Ontario
M9W 5L1

(416) 235-5842 or 235-6055

Ontario Ministry of the Environment
Laboratory Services Branch
LCS-QA/QC Section
125 Resources Rd.
Rexdale, Ontario
M9W 5L1
(416) 235-5842 or 235-6055
FAX (416) 235-5744

May 2, 1989.

TO: PARTICIPANTS OF MOE ROUND ROBIN 89-5

Please find enclosed three (3) 1000 mL amber bottles. The samples are labelled as follows:

EXT A EXT B EXT C

If you are missing any of the above items, please contact us at the above phone number immediately.

Your participation in MOE Round Robin 89-5 is greatly appreciated by the Laboratory Services Branch of the Ministry of the Environment.

As stated in the notification distributed April 12, 1989, samples should be analyzed using the principles and protocols outlined in the MISA general regulation (Ontario Reg. 358/88). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limits for storage were indicated in the advance notification.

To ensure timely release of a summary report, results are to be reported within forty (40) days of reception of the samples. Results will not be accepted after June 16, 1989. Report forms to be used are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact us if there are any problems or questions re the round robin.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842

Catherine Doehler
MISA Audit Scientist
(416) 235-6055

Ontario Ministry of the Environment
Laboratory Services Branch
LCS-QA/QC Section
125 Resources Rd.
Rexdale, Ontario
M9W 5L1

(416) 235-5842

September 15, 1989.

TO: PARTICIPANTS OF MOE INTERLABLABORATORY STUDY 89-5

Thank you for your participation in the MOE Interlaboratory Study 89-5 conducted in May 1989 to assess interlaboratory variability for the analysis of acid and base/neutral extrable organic parameters.

Attached are all the results reported to me as of June 30, 1989. Not all the original participants were able to report results. Listed for each sample and parameter are the expected values, the results reported by each participant, the mean, minium result, maximum result, and the standard deviation. All participants are identified only by their identification code.

A formal report is presently being written. All participants reporting results will receive a copy when it becomes available.

Please contact me if you have any further questions.

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist

Attachment